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TECHNICAL NOTE

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LUBRICATING PROPERTIES OF CERAMIC-BONDED CALCIUM

FLUORIDE COATINGS ON NICKEL-BASE ALLOYS

FROM 75° TO 1900° F

By Harold E. Sliney

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NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

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SUMMARY

The endurance life and the friction coefficient of ceramic-bonded calcium fluoride (CaF_2) coatings on nickel-base alloys were determined at temperatures from 75° to 1900° F. The specimen configuration consisted of a hemispherical rider (3/16-in. rad.) sliding against the flat surface of a rotating disk.

Increasing the ambient temperature (up to 1500° F) or the sliding velocity generally reduced the friction coefficient and improved coating life. Base-metal selection was critical above 1500° F. For instance, cast Inconel sliding against coated Inconel X was lubricated effectively to 1500° F, but at 1600° F severe blistering of the coatings occurred. However, good lubrication and adherence were obtained for René 41 sliding against coated René 41 at temperatures up to 1900° F; no blisters developed, coating wear life was fairly good, and the rider wear rate was significantly lower than for the unlubricated metals. Friction coefficients were 0.12 at 1500° F, 0.15 at 1700° F, and 0.17 at 1800° and 1900° F. Because of its ready availability, Inconel X appears to be the preferred substrate alloy for applications in which the temperature does not exceed 1500° F. René 41 would have to be used in applications involving higher temperatures.

Improved coating life was derived by either preoxidizing the substrate metals prior to the coating application or by applying a very thin (<0.0002 in.) burnished and sintered overlay to the surface of the coating. Preoxidation did not affect the friction coefficient. The overlay generally resulted in a higher friction coefficient than that obtained without the overlay. The combination of both modifications resulted in longer coating life and in friction coefficients intermediate between those obtained with either modification alone.

INTRODUCTION

The lubrication of bearing surfaces at temperatures above about 500° F is a problem area in which solid lubricants are of special interest. Bearings can be lubricated with some soft ceramic or glass coatings that have the interesting property of providing the most effective lubrication when running hot. Examples of this type of coating are the lead monoxide - base (PbO) coatings described in references 1 to 3. At temperatures up to 1250° F, low friction coefficients and low metallic wear were obtained with 440-C and 304 stainless steels lubricated by thin, bonded PbO-base coatings. The coating composition contained about 5 weight percent silicon dioxide (SiO₂) or vanadium pentoxide (V₂O₅). From 75° to 1000° F, low friction was also obtained with 440-C lubricated by PbO coatings containing 5 weight percent boric oxide (B₂O₃), but rider wear was high at 1000° F (ref. 3). It has been reported by others that lead borate glasses (12 and 23.8 percent B₂O₃, balance PbO) provided low friction coefficients when the viscosity of the glasses was less than about 10³ poises (ref. 4). This occurred at about 950° to 1100° F depending on composition. Low friction has also been obtained with molten B₂O₃ above 1000° F (refs. 4 and 5). Lead sulphide (PbS) - B₂O₃ films with friction coefficients less than 0.2 at 1000° F have been reported (ref. 6).

In an effort to develop ceramic coatings which would lubricate above 1250° F, a large number of inorganic compounds were screened at the Lewis Research Center on the basis of their thermodynamic stability, vapor pressure, and other pertinent physical properties (ref. 7). The results indicated that calcium fluoride (CaF₂) would be one of the more promising compounds for further consideration because it is chemically stable in hot oxidizing and reducing atmospheres, has a low vapor pressure, low water solubility, and a crystal structure possessing planes of easy cleavage.

The results of the lubrication studies reported in reference 7 demonstrated that sintered CaF₂ and ceramic-bonded CaF₂ coatings can effectively lubricate alloys such as cast Inconel sliding against Inconel X at temperatures up to at least 1500° F. The results obtained with ceramic-bonded CaF₂ were promising enough to indicate that further study of this type of coating would be in order.

The objective of the research reported herein, therefore, was to conduct a detailed investigation into the effects of sliding velocity, temperature, load, and modifications in coating procedure on the endurance life and the friction properties of CaF₂ coatings.

The lubricating properties of ceramic-bonded CaF_2 on several nickel-base alloys were determined at ambient temperatures from 75° to 1900° F, sliding velocities from 430 to 5200 feet per minute, and loads of 1000 and 2000 grams. The specimen configuration was a $3/16$ -inch-radius hemisphere sliding against the flat surface of a rotating disk.

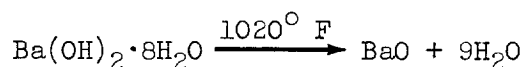
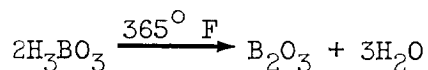
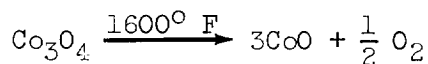
Modifications in coating procedures studied were preoxidation of the base metal, treatment of the coating after application to the metal, and control of coating thickness.

FORMULATION AND APPLICATION OF COATINGS

Formulation

As described in detail in reference 7, the ceramic binder was formulated on the basis of melting point (or softening range), vitrifying tendency, and thermal expansion properties.

The raw materials were reagent grades of cobalt oxide (Co_3O_4), boric acid (H_3BO_3), and barium hydroxide octahydrate ($\text{Ba}(\text{OH})_2 \cdot 8\text{H}_2\text{O}$). References 8 and 9 indicate that these compounds will decompose to CoO , B_2O_3 , and BaO , respectively, at the temperatures indicated in the following assumed overall decomposition reactions:



Coating Procedure

The coating procedure described in reference 7 has been modified. The following stepwise procedure yielded the best results:

(1) A mixture of Co_3O_4 , H_3BO_3 , and $\text{Ba}(\text{OH})_2 \cdot 8\text{H}_2\text{O}$ was prepared with the following weight percentages: 45.6 percent Co_3O_4 , 25.3 percent H_3BO_3 , and 29.1 percent $\text{Ba}(\text{OH})_2 \cdot 8\text{H}_2\text{O}$.

(2) The mixture was melted in a porcelain crucible at 2200° F until reactions ceased and a uniform, quiescent melt was obtained. The calculated composition after completion of the decomposition reactions was 60 percent CoO, 20 percent B_2O_3 , and 20 percent BaO.

(3) The melt was poured slowly into cold water to form friable shot-like globules, which were then filtered, dried, and ground to pass through a 200-mesh screen.

(4) The resulting frit was mixed with CaF_2 (25 wt. percent frit, 75 wt. percent CaF_2), and then pebble-milled to pass through a 300-mesh screen.

(5) The mixture was then stirred into an aqueous slurry with a high-speed blender. Water content was not critical; the slurry was made dilute enough to prevent clogging of spray equipment but not so dilute as to allow rapid separation of a water layer.

(6) The metal specimens were preoxidized by heating in air to 2000° F until a deep blue oxide film formed. This is an interference color corresponding to a film thickness of about 700 angstroms.

(7) The specimens were allowed to cool to about 300° F and then sprayed, preferably with an airbrush. The water immediately evaporated, and the thin solid film that was left was built up to the desired thickness by repeated passes of the spray.

(8) The specimens were fired at 2000° F until the binder was molten and then slowly air-cooled. Cooling from 2000° to 900° F over about a 2-minute interval was accomplished by withdrawal of the specimen from the hot zone of the furnace at a controlled rate.

(9) The specimens were burnished with fine steel wool or fine garnet paper. Final coating thickness was between 0.001 and 0.002 inch.

(10) When an overlay was used, wet CaF_2 was burnished into the surface of the coating. The specimen was heated to about 180° F during this process to hasten evaporation of the water. The overlay was then sintered at 1700° F. For best results burnishing and sintering were carried out twice. This treatment did not increase overall coating thickness by more than 0.0002 inch.

EXPERIMENTAL APPARATUS AND PROCEDURE

The apparatus used in performing the lubrication studies is shown in figure 1. A detailed description is given in reference 8. Basically

E-1401

the apparatus consists of a rotating disk placed in sliding contact with a hemispherically tipped rider (3/16-in.-rad. hemisphere), usually under a normal load of 1000 grams. The rider describes a 2-inch-diameter wear track on the disk. Sliding is unidirectional at controlled velocities up to 1260 feet per minute. For higher speeds or for temperatures above 1600° F, the induction-heated rig shown in figure 1(b) is used. Friction torque is measured with strain gages and continuously recorded. Most specimens were "run-in" with incrementally increased loads according to the following procedure: 2 minutes at 200 grams, 2 minutes at 400 grams, 2 minutes at 600 grams, 2 minutes at 800 grams, and, finally, 1000 grams for the balance of the test. To study the effect of high initial loads, others were run-in at 3000 grams for 2 minutes followed by 2 minutes at 400, 600, and 800 grams.

Before each test, the rider and disk specimens were cleaned as follows:

- (1) Washed with acetone
- (2) Scrubbed with levigated alumina (omitted when cleaning coated disks to avoid embedding alumina particles into coatings)
- (3) Rinsed with hot tap water
- (4) Rinsed briefly with distilled water
- (5) Blotted dry with filter paper
- (6) Stored in desiccator

RESULTS AND DISCUSSION

The results of this study showed that the selection of the base metal and variations in coating procedure had an important influence on the lubricating properties of ceramic-bonded CaF_2 coatings. The results also demonstrated the influence of ambient test temperature, sliding velocity, and load.

Influence of Calcium Fluoride Overlay

The data shown in figure 2(a) indicate that a very thin (0.0002 in.) burnished and sintered overlay of CaF_2 on the coating surface was beneficial in improving coating life. However, below 1500° F the friction coefficients were higher for coatings with a CaF_2 overlay. The endurance of coatings with an overlay was determined

at loads of 1000 and 2000 grams. Load did not clearly influence coating life, but lower friction coefficients were obtained at the higher load.

Influence of Base-Metal Preoxidation

Oxidation of the substrate metal before spraying and firing of the coatings resulted in improved coating wear life and had no detrimental influences on the friction coefficient (fig. 2(b)). As indicated in figure 2(b), some of the specimens were run-in at a high load (3000 g for 2 min) instead of the usual light loads. The data suggest that high initial loads are beneficial to coating life. During friction and endurance runs, quiet running and a steady friction torque were obtained almost immediately at 3000 grams, but an initial period of erratic friction occurred at 1000 grams. When the load was reduced from 3000 to 1000 grams, the specimens continued to run quietly with a steady friction torque. For coatings with no overlay, the steady-state friction coefficient was not appreciably influenced by load.

Influence of Combined Preoxidation and

Calcium Fluoride Overlay

Data illustrating the combined effect of base-metal preoxidation and the CaF_2 overlay are given in figure 2(c). This combined treatment resulted in an increase in coating life which was superior to that resulting from the CaF_2 overlay or preoxidation alone. However, the friction coefficients were intermediate between those of the coated specimens modified only with a CaF_2 overlay and those modified only by preoxidation of the base metal. A summary of the data of figures 2(a), (b), and (c) is given in figure 2(d).

Endurance of Resin-Bonded Molybdenum Disulfide and

Lead Oxide Coatings

Resin-bonded molybdenum disulfide (MoS_2) coatings have become accepted lubricants in many applications at temperatures up to about 500° F. Lead oxide coatings have demonstrated good endurance properties in ball-bearing tests at 1000° and 1250° F (ref. 2). Therefore, in figure 3 the endurance of CaF_2 coatings is compared with that of MoS_2 and PbO coatings.

The endurance life of a CaF_2 coating at any given temperature was considered good if it were of about the same order of magnitude as that of MoS_2 at temperatures between 75° and 500° F or of PbO at 1000° F. On this basis, ceramic-bonded CaF_2 coatings on preoxidized Inconel X should have adequate endurance for many applications between 500° and 1500° F.

Effect of Coating Thickness

The friction coefficients of dry film lubricants are usually influenced by the thickness of the film. In general, the most favorable results are obtained with thin films (0.0002 in. for MoS_2 and 0.0010 in. for PbO). However, coating thickness had no appreciable effect on the friction coefficient of ceramic-bonded CaF_2 for coating thicknesses between 0.0005 and 0.0030 inch. On the other hand, overlay thickness was important. The effect of overlay thickness on the friction coefficient at 1500° F is shown in figure 4. Without an overlay, the friction coefficient was 0.15. Enrichment of the surface with burnished and sintered CaF_2 surface films of not more than 0.0005-inch thickness reduced the friction coefficient to between 0.06 and 0.09. With an overlay thickness greater than 0.0005 inch, the friction coefficient was about 0.2. Below 1500° F, even very thin overlays resulted in increased friction.

Effect of Sliding Velocity

Increases in sliding velocity cause a rise in the frictional heat generated and consequently a rise in surface temperature. The data illustrating the effect of temperature have shown that ceramic-bonded CaF_2 lubricates best at high temperatures. It seems likely, therefore, that effective lubrication should be obtained at lower ambient temperatures when the sliding velocity is increased. That this is actually the case is shown in figures 5 and 6.

In figure 5, the friction coefficient of ceramic-bonded CaF_2 with an overlay is shown as a function of sliding velocity and temperature. At 75° F, the friction coefficient decreased from 0.32 at 430 feet per minute to 0.19 at 5200 feet per minute. At 1000° and 1500° F, only a slight decrease in friction coefficient was obtained when the velocity was increased from 430 to 1000 feet per minute, and essentially no change was observed with further increase in velocity. At sliding velocities greater than 1000 feet per minute, the friction coefficient at 1000° F was about 0.15, and at 1500° F it was about 0.06.

At 1260 feet per minute and 750° F, the coating life was about equal to the life at 430 feet per minute and 750° F (fig. 6). At 1260 feet per minute, the friction coefficients were equal to or less than 0.2 from 750° to 1500° F. A friction coefficient of 0.2 is customarily considered to be the upper limit for acceptable lubrication with solid lubricants intended for use under severe sliding conditions.

Influence of Ceramic-Bonded Calcium Fluoride in Reducing Metallic Wear

In figure 7(a), the wear rates at 1000° F for uncoated cast Inconel sliding against unlubricated Inconel X and against CaF₂-lubricated Inconel X are compared. The rider wear rates were constant in both cases and much lower wear was obtained with CaF₂-lubricated specimens. Photographs and surface profiles of the specimens are shown in figure 7(b). The wear surfaces of the lubricated specimen are smooth and glazed. There is no evidence of galling or metal transfer. For the unlubricated specimen, the evidence of severe galling and metal transfer is unmistakable. Surface profiles were taken across the wear tracks of the disk specimens with a stylus-type surface analyzer. An extremely smooth surface was apparent on the wear track of the CaF₂-lubricated disk. On the unlubricated disk, metal transfer from the cast Inconel rider resulted in deposits, some of which were greater than 0.0010 inch in height. Pulled-out areas, typically 0.0002 to 0.0004 inch deep were also in evidence on the Inconel X surface.

The constant wear rates indicated in figure 7(a) were characteristic of relatively short duration experiments. During prolonged endurance runs, a decrease in wear rate was observed. Typical results are shown in figure 8. A constant wear rate existed from 1 to 3 hours. Then a gradual decrease in rate occurred to a value which then remained constant for the remaining life of the coating.

Lubricating Properties of Ceramic-Bonded

Calcium Fluoride Above 1500° F

Calcium fluoride, which melts at 2480° F, has no inherent property that should limit its usefulness as a solid lubricant to 1500° F. Rather, the temperature limitation is imposed by the low mechanical strengths or the appreciable oxidation rates of most alloys above 1500° F. The refractory metals (tungsten, molybdenum, and columbium) were considered outside the scope of this investigation for the present because they require protective atmospheres at elevated temperatures to prevent severe

oxidation (ref. 9). The high-temperature nickel-base alloys as a group have yield and creep strengths and oxidation resistance superior to the cobalt-base alloys (ref. 10). Therefore, experiments were performed to determine the maximum temperature at which nickel-base alloys, including Inconel X, might possibly be lubricated in air with ceramic-bonded CaF_2 .

The yield strengths from 75° to 1800° F of alloys used in this investigation are given in figure 9. The curves were plotted from data found in reference 11. The data indicate that René 41 retains useful strength to higher temperatures than the other alloys.

Coatings were applied to René 41, Hastelloy C, and Inconel X. The nominal compositions of these alloys are given in table I. The lubricating properties of the coatings were determined at temperatures to 1600° F on Inconel X and Hastelloy C and to 1900° F on René 41. Friction coefficients and coating lives are given in table II. For coated Hastelloy C at 1500° F, the friction coefficient was satisfactory (0.15), but coating wear life was poor; at 1600° F, severe spalling of the coating occurred (fig. 10). For coated Inconel X (no overlay) at 1500° F, the friction coefficient was 0.15, and coating wear life was good; at 1600° F, the friction coefficient was excellent (0.08) and wear life was good, but coated areas outside the wear track were blistered (fig. 10). Up to 1900° F, CaF_2 lubricated René 41 effectively ($f = 0.12$ to 0.17). Endurance life was good to 1700° F and fair at 1800° and 1900° F. No apparent damage to coating adhesion occurred at any temperature (fig. 10).

The far better adhesion of the coatings on René 41 above 1500° F may possibly be attributed to the fact that René 41 is vacuum-melted and the other alloys are air-melted. Air-melted alloys have a higher concentration of dissolved gases and other impurities which might have a detrimental influence on coating adhesion at high temperatures.

In figure 11 the friction and wear of unlubricated Inconel X and René 41 are compared with the friction and wear of these metals when lubricated with a CaF_2 coating. The friction and wear of cast Inconel on unlubricated Inconel X at 1500° and 1600° F are higher than the corresponding friction and wear of René 41 at 1500° , 1800° , or 1900° F. However, when the same metal combinations were lubricated with CaF_2 , only slightly better results were obtained with René 41. Of course, the loss of coating adhesion on Inconel X at 1600° F precludes its use above 1500° F.

At 1700° F, rider wear with CaF_2 -lubricated René 41 was very low, and the friction coefficient was 0.15. At 1800° and 1900° F, the friction coefficient was 0.17. Coating life was good and metallic wear was fair.

The friction and wear of unlubricated René 41 in air and in a dry reducing atmosphere (10 percent hydrogen, 90 percent nitrogen) were compared at 1700° F. The resulting wear scar and tracks are shown in figure 12. A fairly smooth, glazed oxide film covers most of the wear surface area of the specimens run in air. In the reducing atmosphere, severe galling, metal transfer, and plastic deformation occurred. The friction coefficient was 0.4 compared with 0.23 in air.

The ability of nickel-chromium alloys to form oxide films which are, to some extent, self-lubricating is a desirable characteristic when they are used as substrates for dry film lubricants. Small failure areas in the coating do not rapidly lead to catastrophic failure. The low rate of surface damage allows time for the lubricant film to repair itself by plastic flow from unfailed areas, or at least prolongs the time before the friction coefficient increases to a value considered to be a failure condition.

SUMMARY OF RESULTS

The lubricating properties of ceramic-bonded CaF_2 were determined at temperatures up to 1900° F. At any given temperature, lubrication was considered effective when the friction coefficient was 0.2 or less, galling of the metal specimens was prevented, and the wear life of the coating was of the same order as that obtained from resin-bonded molybdenum disulfide at 75° F. The major results were:

1. A vacuum-melted, nickel-base alloy (René 41) was lubricated effectively to 1900° F. For air-melted, nickel-base alloys with similar composition, lubrication was effective to 1500° F, but coating adhesion was unsatisfactory at higher temperature.
2. Oxidation of the base metal before coating application was beneficial to coating wear life and generally resulted in a more uniform, more adherent coating than was obtained when this step was omitted.
3. A very thin calcium fluoride overlay burnished and sintered onto the base coating resulted in improved endurance life but also resulted in a higher friction coefficient at all temperatures below about 1400° F.
4. Typical of solid lubricants containing a ceramic phase, the friction coefficients and coating wear life were most favorable under conditions that were conducive to high surface temperatures, such as high ambient temperatures, high sliding velocities, and high load.
5. At light loads (100 to 1000 g), the friction coefficient was often erratic for a short initial period of time; at high loads (2000 to 3000 g), the friction coefficient immediately assumed a steady value.

Increasing the load resulted in lower friction coefficients for coatings with a calcium fluoride overlay, but had no effect on the steady-state friction coefficient of coatings without an overlay.

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National Aeronautics and Space Administration
Cleveland, Ohio, November 9, 1961

E-1401

APPENDIX - CONDUCTIVITY MEASUREMENTS FOR STUDYING FAILURE OF SOLID LUBRICANT FILMS

There is considerable background for the use of electrical resistance measurements to study the failure of liquid lubricant films (e.g., refs. 12 and 13). However, no reference has been found which describes the use of electrical measurements to provide a continuous indication of the extent and distribution of failure areas in solid lubricant films.

The circuit employed for the latter purpose is shown in figure 13. A small direct-current voltage E is placed in series with two equal resistors R_1 and R_2 . The test specimens are insulated so that electrical contact between them can occur only at the sliding surfaces. The contact resistance R_3 between the friction specimens is in parallel with R_2 . An oscilloscope continuously measures the voltage drop V across R_1 .

When the contact resistance R_3 is very high compared with R_2 , the voltage across R_2 is nearly equal to the voltage across R_1 , and, to a good approximation, $V = E/2$. When R_3 is very low, as when coating failure results in metallic contact, the current bypasses R_2 , and, to a good approximation, $V = E$. Therefore, V can vary between $E/2$ and E . The oscilloscope amplitude is adjusted to give full-scale deflection at $V = E$ and a base line at $V = E/2$.

When the sweep frequency of the oscilloscope is synchronized with the rotational frequency (in rpm) of the drive shaft, the resulting pattern displayed on the oscilloscope is a visual analog of the distribution of contact resistance along the circumference of the wear track.

A network analysis of the circuit in figure 13 gives the following relation:

$$\frac{R_3}{R_2} = \frac{1 - V/E}{2V/E - 1} \quad (1)$$

The ratio R_3/R_2 is plotted as a function of V/E in figure 13. The sensitivity of the measurements is readily controlled by properly matching the fixed resistances R_2 and R_1 with the initial contact resistance R_3^0 . As a coating wears, R_3/R_2 decreases from the initial value selected to nearly zero when metal-to-metal contact occurs. The value of R_2 , therefore, determines the portion of the curve in figure 13 over which measurements will be made.

If contact resistance R_3 is assumed to be linearly proportional to coating thickness, coating thickness can be plotted against oscilloscope deflection for various values of R_3^0/R_2 , as shown in figure 14. For $R_3^0/R_2 = 100$, almost no deflection occurs until metal-to-metal contact occurs. For $R_3^0/R_2 = 10$, the deflection increases gradually as the coating wears and shows a sharp increase when only a very thin film remains. For $R_3^0/R_2 = 1$, the deflection increases almost linearly with decreasing coating thickness. For $R_3^0/R_2 = 0.1$, the measurements become too sensitive to minor variations in resistance even on thick coatings.

One error in assuming that the peaks on the oscilloscope trace are only a function of coating thickness is apparent in figure 15. Completely failed areas caused deflection of only 50 percent of full scale. This is probably due to the electrical resistance of an oxide film which is present as an intermediate layer between the base metal and the coating.

This technique is quite flexible and is capable not only of indicating coating failure but also of giving a continuous indication of the approximate coating-thickness distribution in the wear track at any time during determination of coating endurance life. Some examples of the oscilloscope patterns obtained during progressive stages of coating failure are shown in figures 15 to 17.

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E-1401

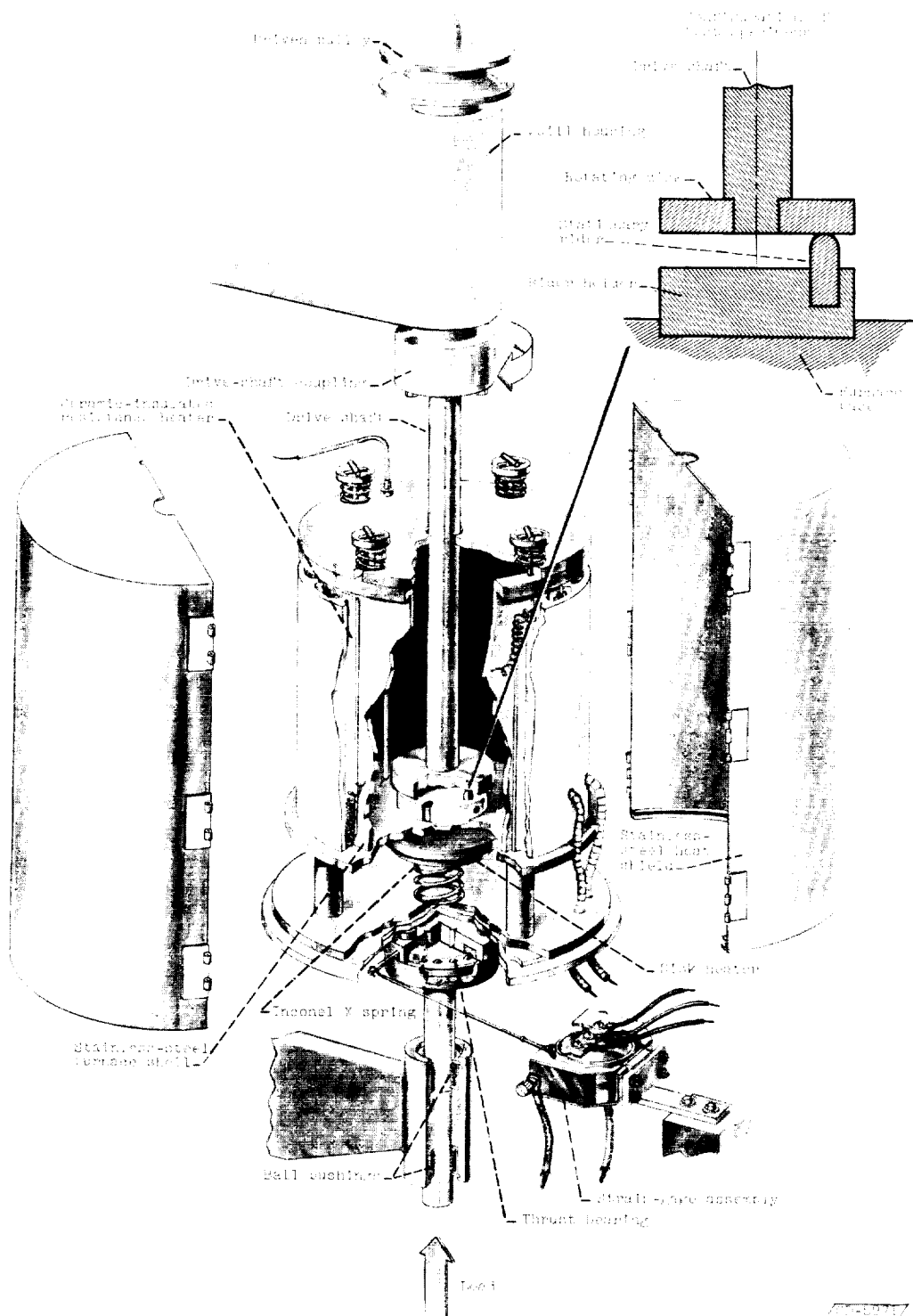
TABLE I. - NOMINAL COMPOSITION OF ALLOYS USED IN THIS INVESTIGATION

Alloy	Chemical composition, weight percent												
	Ni	Co	Cr	Fe	C	Si	Mo	Al	Mn	Cu	Nb	Ti	W
Cast Inconel	Balance	----	13.5	6	0.2	2	----	---	0.8	0.25	-	---	---
Inconel X	Balance	----	15	7	.4	.4	----	0.7	.5	.2	1	2.5	---
Hastelloy C	Balance	2.5	15	5	.08	1.0	16	---	1.0	----	-	---	3.0
René 41	Balance	11	19	5	.12	.5	9.5	1.5	.1	----	-	3.0	---

TABLE II. - LUBRICATING PROPERTIES OF CERAMIC-BONDED CaF_2 FOR
VARIOUS METAL COMBINATIONS FROM 1500° TO 1900° F

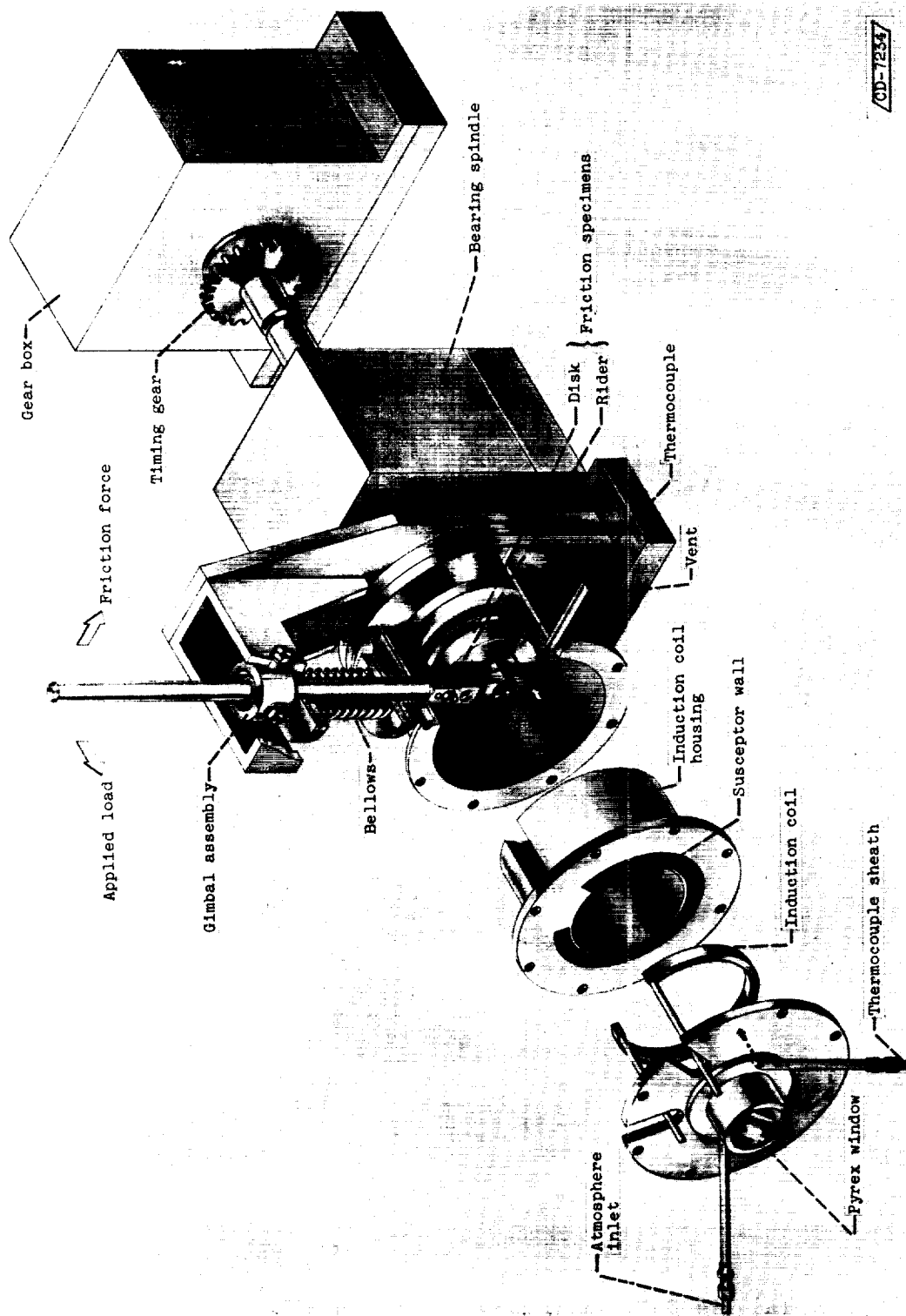
[Atmosphere, air; load, 1000 g; sliding velocity,
600 ft/min; coating thickness, 0.001 to
0.002 in.]

Rider alloy	Disk alloy	Temperature, $^\circ\text{F}$	Friction coef- ficient	Coating life, cycles to failure	Adherence of coating to disk after test
Cast Inconel	Hastelloy C	1500	0.15	1.5×10^2	Good
		1600	.15	3.7×10^4	Coating spalled (probably on cooling)
	Inconel X	1500	0.15	1.0×10^5	Good
		1600	.08	2.0×10^5	Coating badly blistered
René 41	René 41	1500	0.12	2.7×10^5	Good ↓
		1700	.15	3.5×10^5	
		1800	.17	6.0×10^4	
		1900	.17	6.6×10^4	



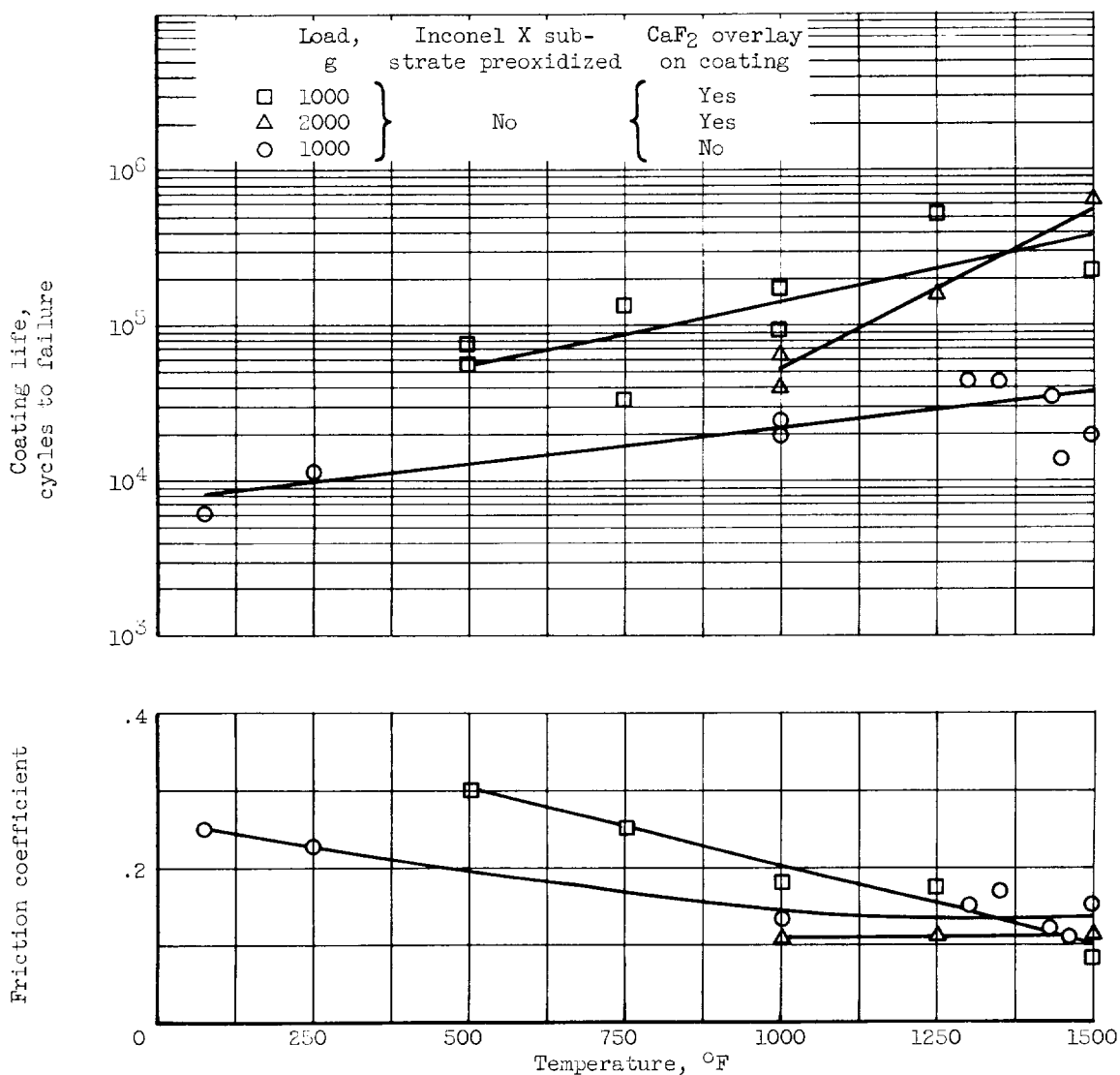
(a) Rig for temperatures up to 1500° F.

Figure 1. - Apparatus for lubrication studies.



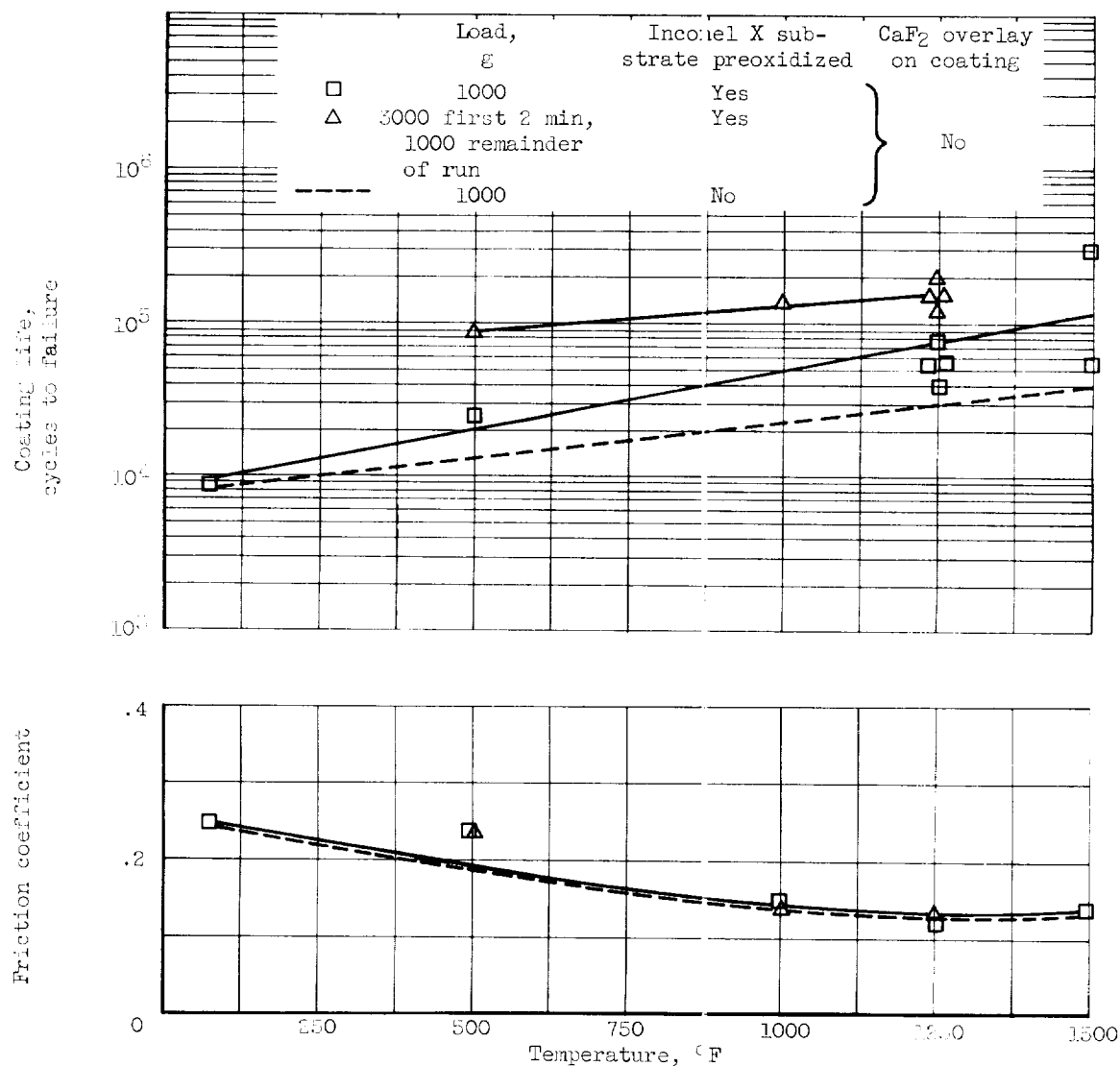
(b) Induction-heated rig for temperatures up to 2000° F.

Figure 1. - Concluded. Apparatus for lubrication studies.



(a) Influence of calcium fluoride overlay and load.

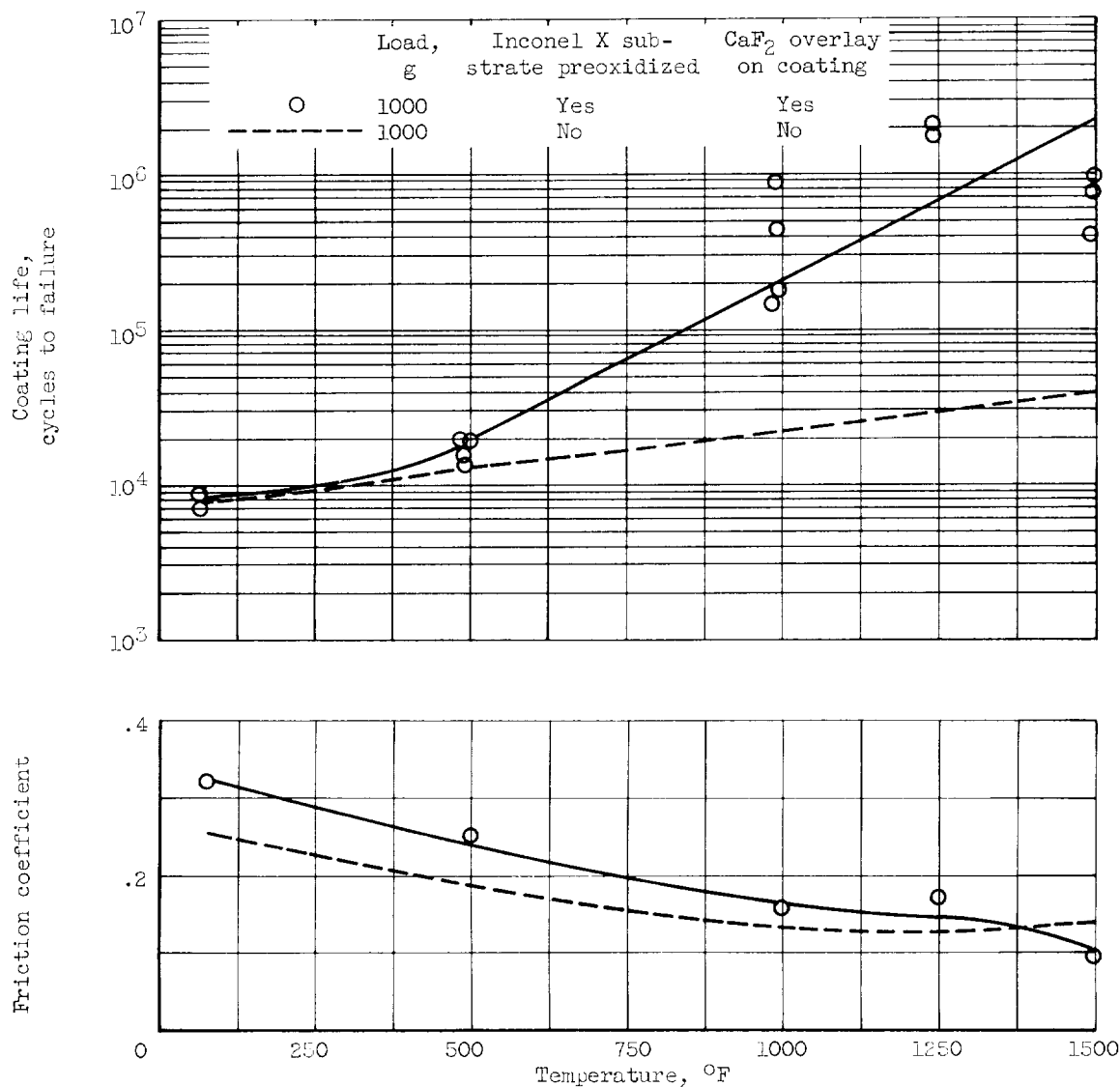
Figure 2. - Effect of temperature and modifications in coating procedure on lubricating properties of ceramic-bonded calcium fluoride coatings. Lubricant, 0.001- to 0.002-inch coatings of calcium fluoride with 25 weight percent ceramic binder; binder composition, 60 weight percent CoO, 20 weight percent B₂O₃, 20 weight percent BaO; rider specimens, cast Inconel (3/16-in.-rad. hemispheres); sliding velocity, 430 feet per minute.



(b) Influence of preoxidation of Inconel X.

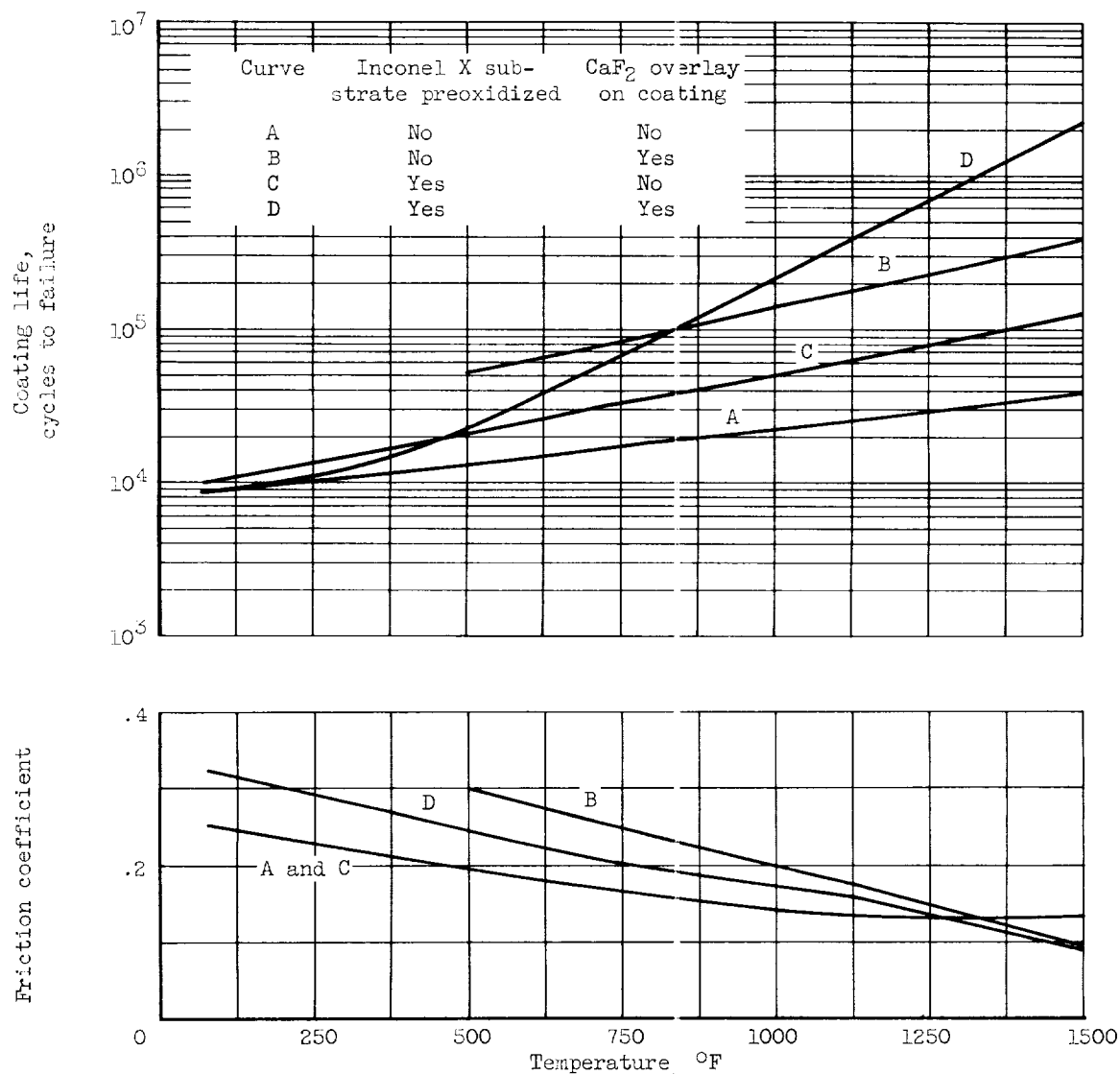
Figure 2. - Continued. Effect of temperature and modifications in coating procedure on lubricating properties of ceramic-bonded calcium fluoride coatings. Lubricant, 0.001- to 0.002-inch coatings of calcium fluoride with 85 weight percent ceramic binder; binder composition, 80 weight percent CoO, 20 weight percent B₂O₃, 20 weight percent BaO; rider specimens, cast Inconel (3/16-in.-rad. hemispheres); sliding velocity, 400 feet per minute.

E-1401



(c) Influence of calcium fluoride overlay combined with preoxidation of substrate metal.

Figure 2. - Continued. Effect of temperature and modifications in coating procedure on lubricating properties of ceramic-bonded calcium fluoride coatings. Lubricant, 0.001- to 0.002-inch coatings of calcium fluoride with 25 weight percent ceramic binder; binder composition, 60 weight percent CoO, 20 weight percent B₂O₃, 20 weight percent BaO; rider specimens, cast Inconel (3/16-in.-rad. hemispheres); sliding velocity, 430 feet per minute.



(d) Summary.

Figure 2. - Concluded. Effect of temperature and modifications in coating procedure on lubricating properties of ceramic-bonded calcium fluoride coatings. Lubricant, 0.001- to 0.002-inch coatings of calcium fluoride with 25 weight percent ceramic binder; binder composition, 60 weight percent CoO, 20 weight percent B₂O₃, 20 weight percent BaO; rider specimens, cast Inconel (3/16-in.-rad. hemispheres); sliding velocity, 430 feet per minute.

E-1401

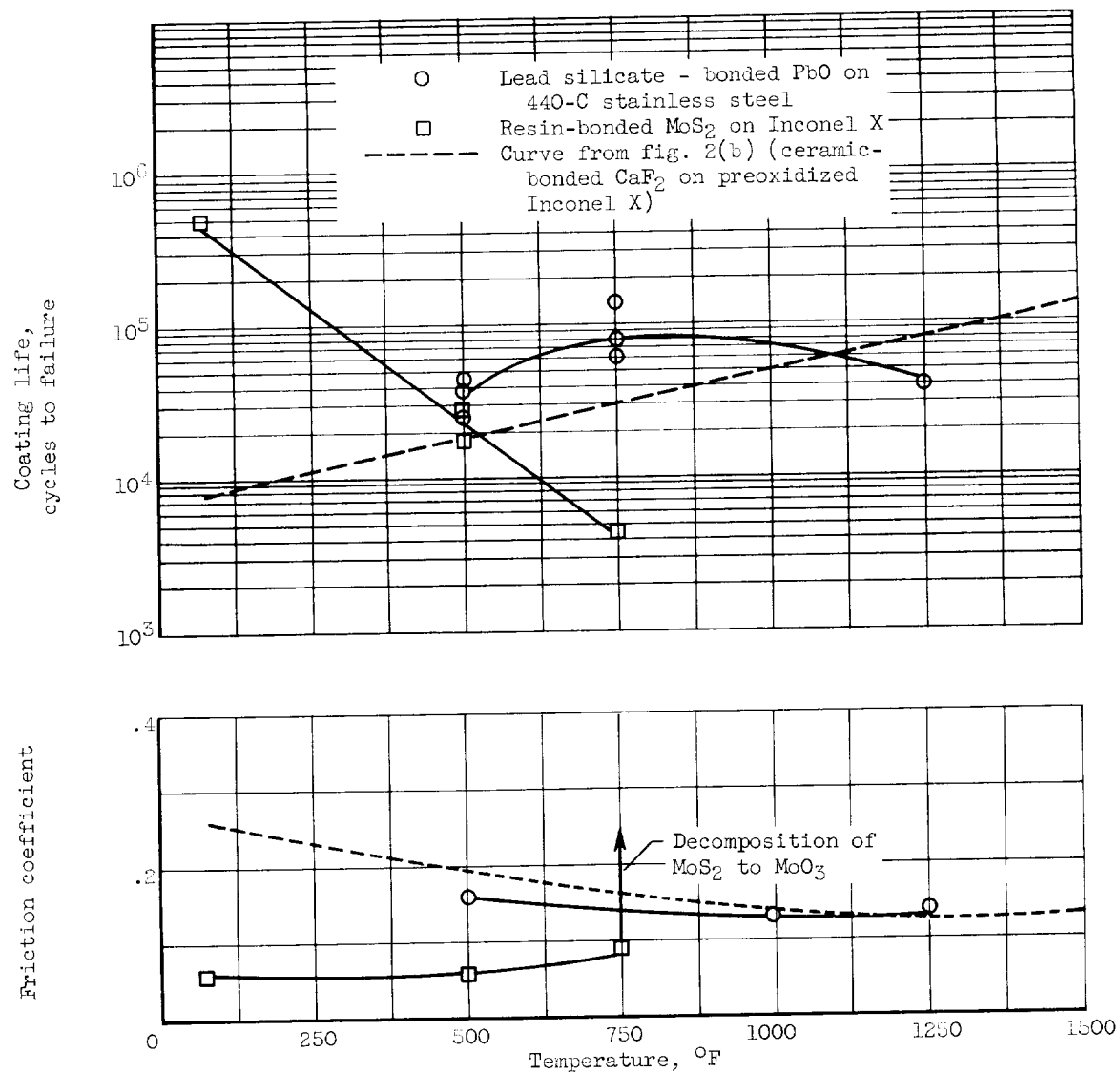


Figure 3. - Effect of temperature on resin-bonded molybdenum disulfide, silicate-bonded lead oxide, and ceramic-bonded calcium fluoride solid lubricant coatings. Rider specimens, cast Inconel (3/16-in.-rad. hemispheres); sliding velocity, 430 feet per minute; load, 1000 grams.

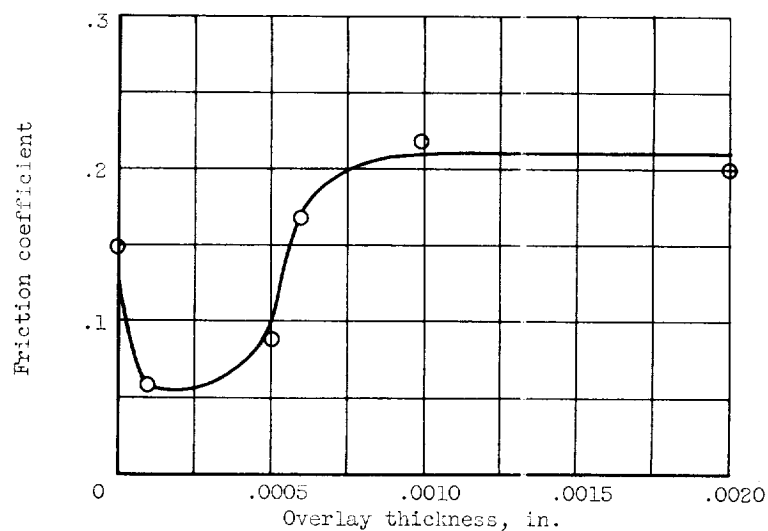


Figure 4. - Influence of thickness of calcium fluoride overlay on friction coefficient of ceramic-bonded calcium fluoride coatings. Temperature, 1400° F; rider specimens, cast Inconel (3/16-in.-rad. hemispheres); substrate metal, Inconel X; sliding velocity, 430 feet per minute; load, 1000 grams.

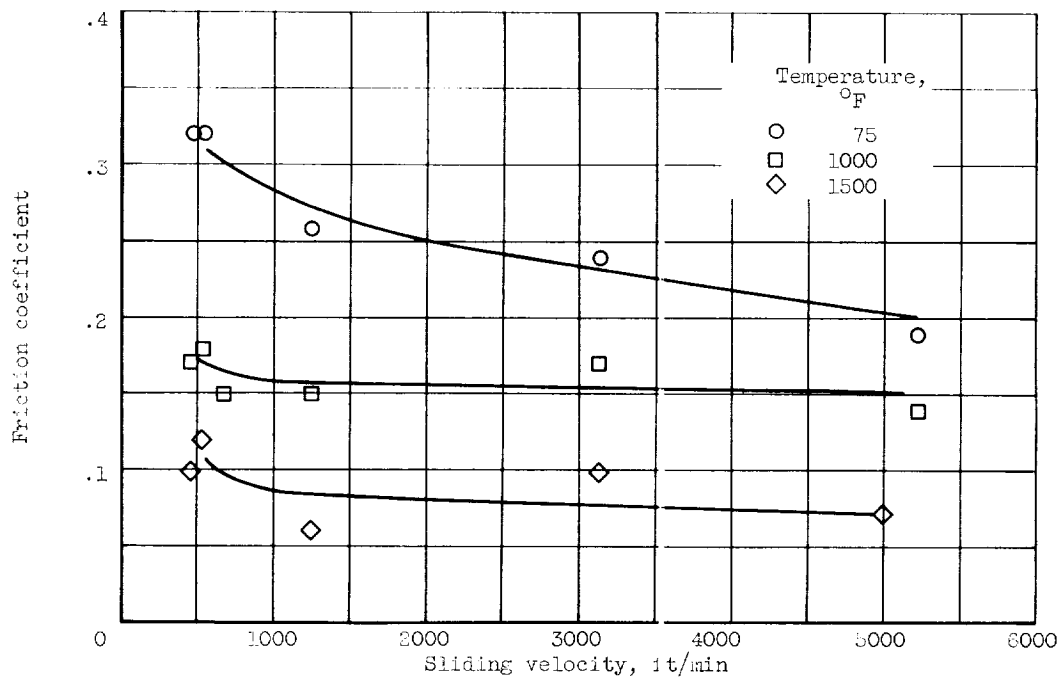


Figure 5. - Effect of sliding velocity on friction coefficient of ceramic-bonded calcium fluoride at various temperatures. Coatings bonded to preoxidized Inconel X, and enriched with burnished and sintered calcium fluoride overlay; rider specimens, cast Inconel (3/16-in.-rad. hemispheres); load, 1000 grams.

E-1401

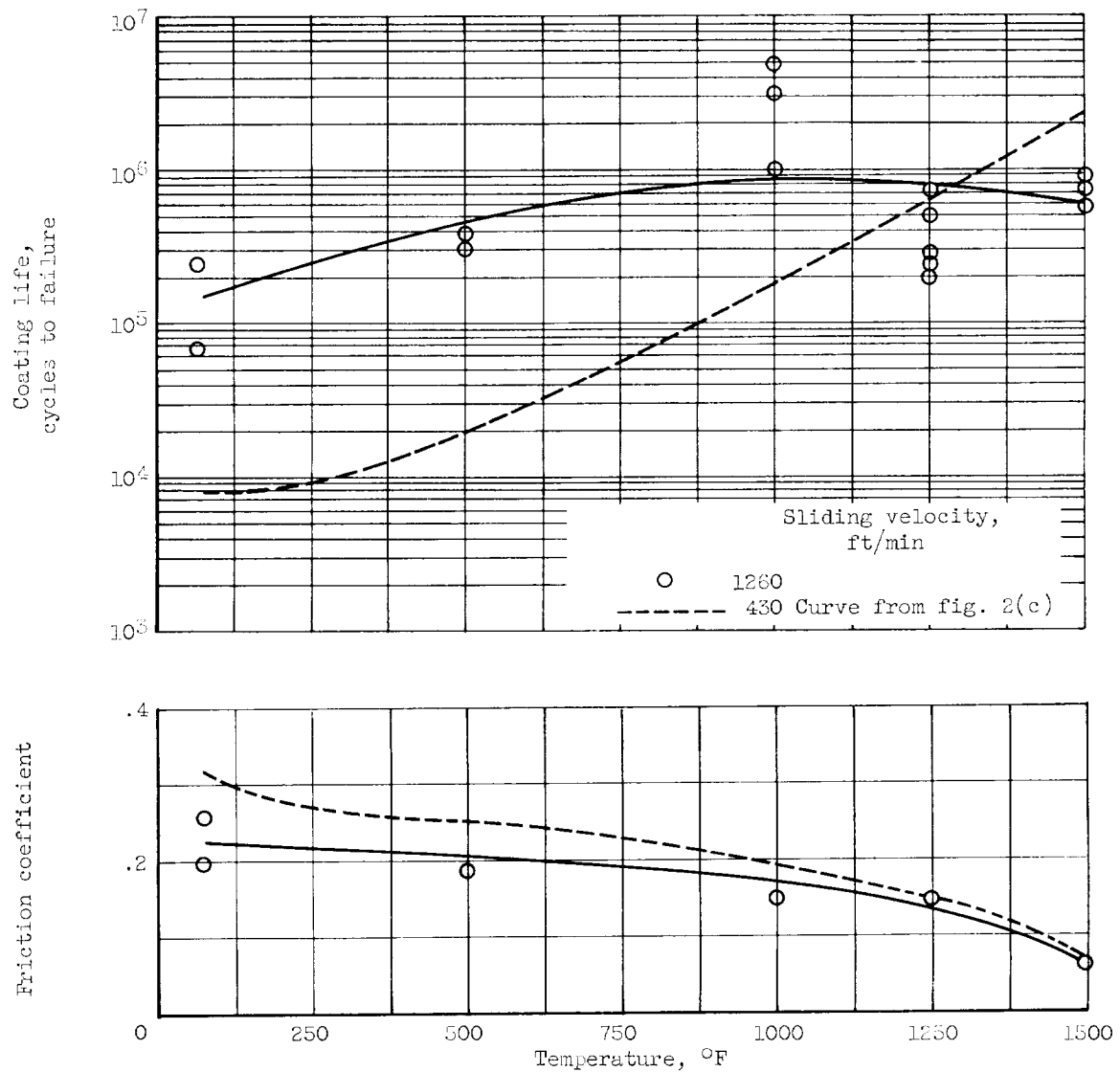
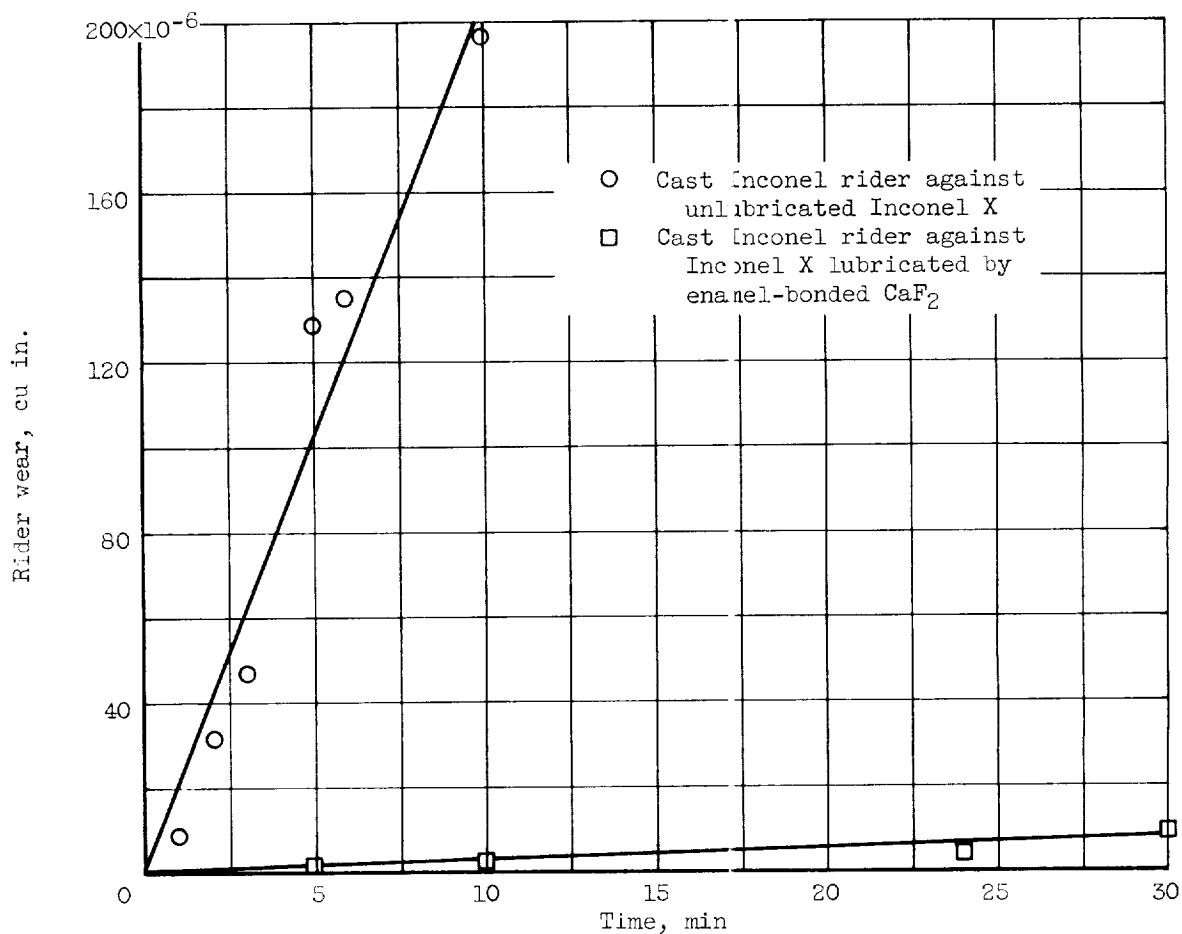


Figure 6. - Lubricating properties of ceramic-bonded calcium fluoride at two sliding velocities. Coatings bonded to preoxidized Inconel X and enriched with burnished and sintered calcium fluoride overlay. Rider specimens, cast Inconel (3/16-in.-rad. hemispheres); load, 1000 grams.



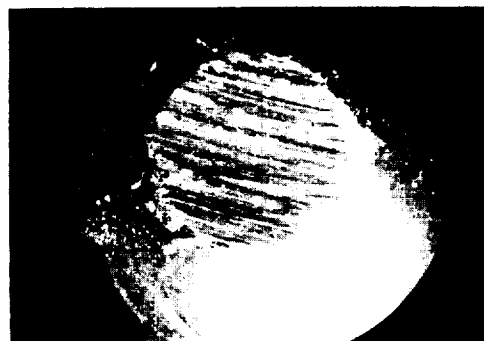
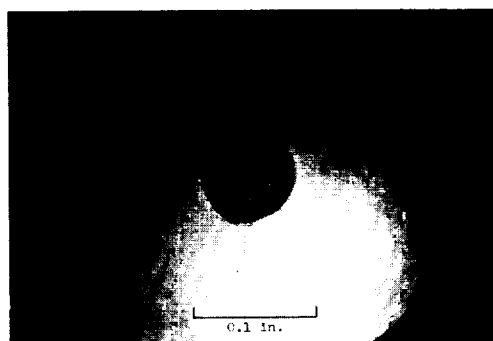
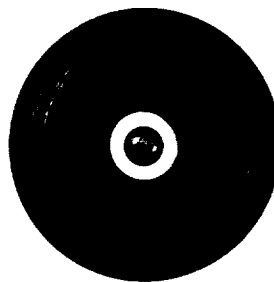
(a) Comparison of wear rates.

Figure 7. - Effectiveness of ceramic-bonded calcium fluoride lubricant in reducing wear. Coating bonded to preoxidized Inconel X and enriched with burnished and sintered calcium fluoride overlay; temperature, 1000°F ; sliding velocity, 430 feet per minute; load, 1000 grams.

E-1401

With CaF_2 lubrication

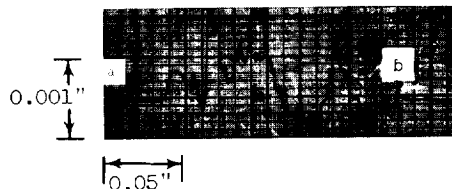
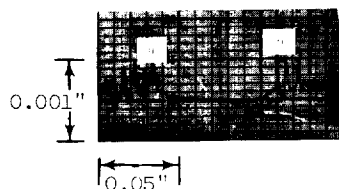
Unlubricated



Wear scars on riders



Wear tracks on disks



Surface profiles across wear tracks

(b) Photographs and surface profiles. Lubricated specimens after 30 minutes; unlubricated specimens after 10 minutes.

Figure 7. - Concluded. Effectiveness of ceramic-bonded calcium fluoride lubricant in reducing wear. Coating bonded to preoxidized Inconel X and enriched with burnished and sintered calcium fluoride overlay; temperature, 1000°F ; sliding velocity, 430 feet per minute; load, 1000 grams.

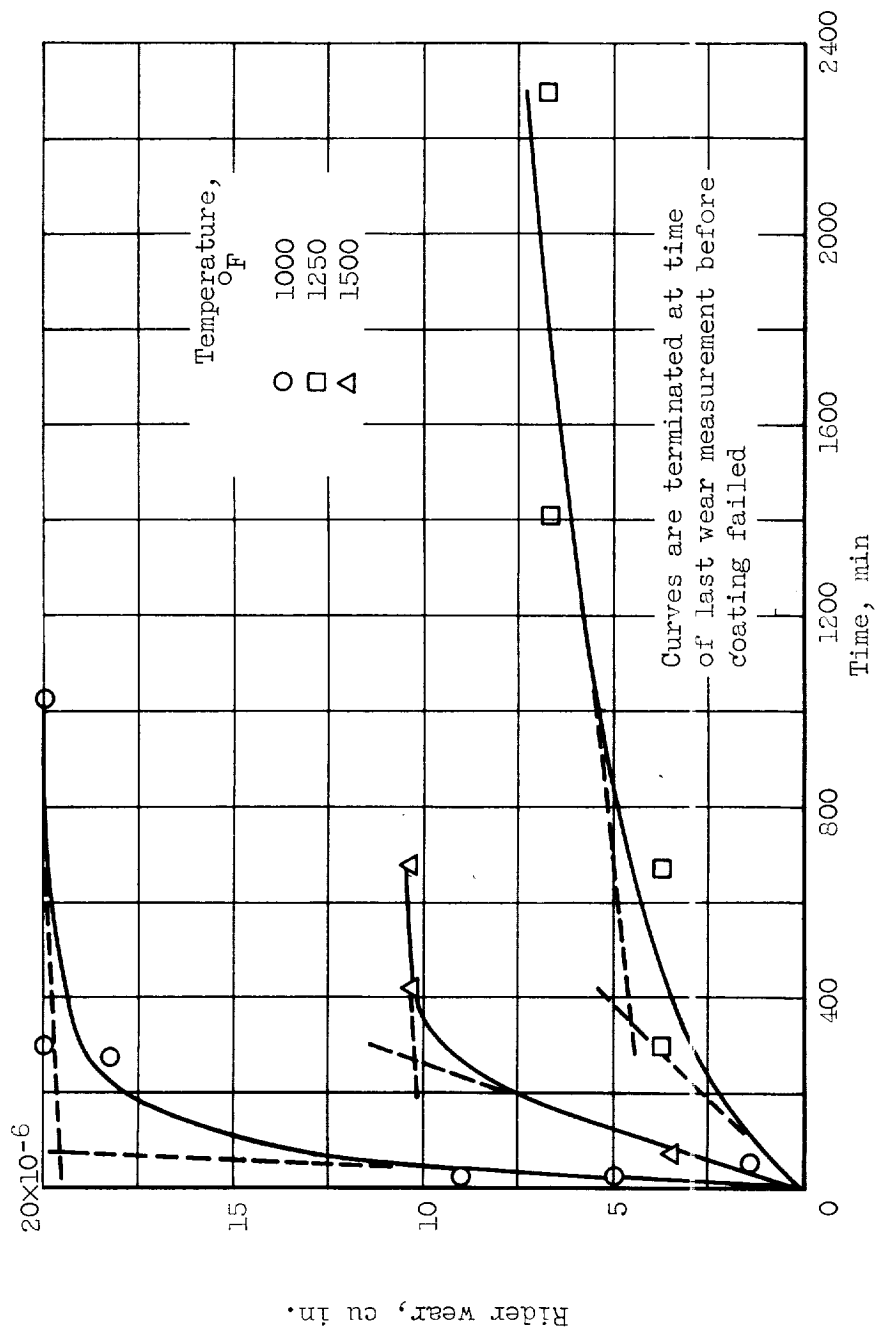


Figure 8. - Wear of cast Inconel rider specimens (3/16-in.-rad. hemispheres) after running at various temperatures against lubricated Inconel X. Coatings enamel-bonded to preoxidized Inconel X and enriched with bur-nished and sintered calcium fluoride overlay. Sliding velocity, 430 feet per minute; load, 1000 grams.

E-1401

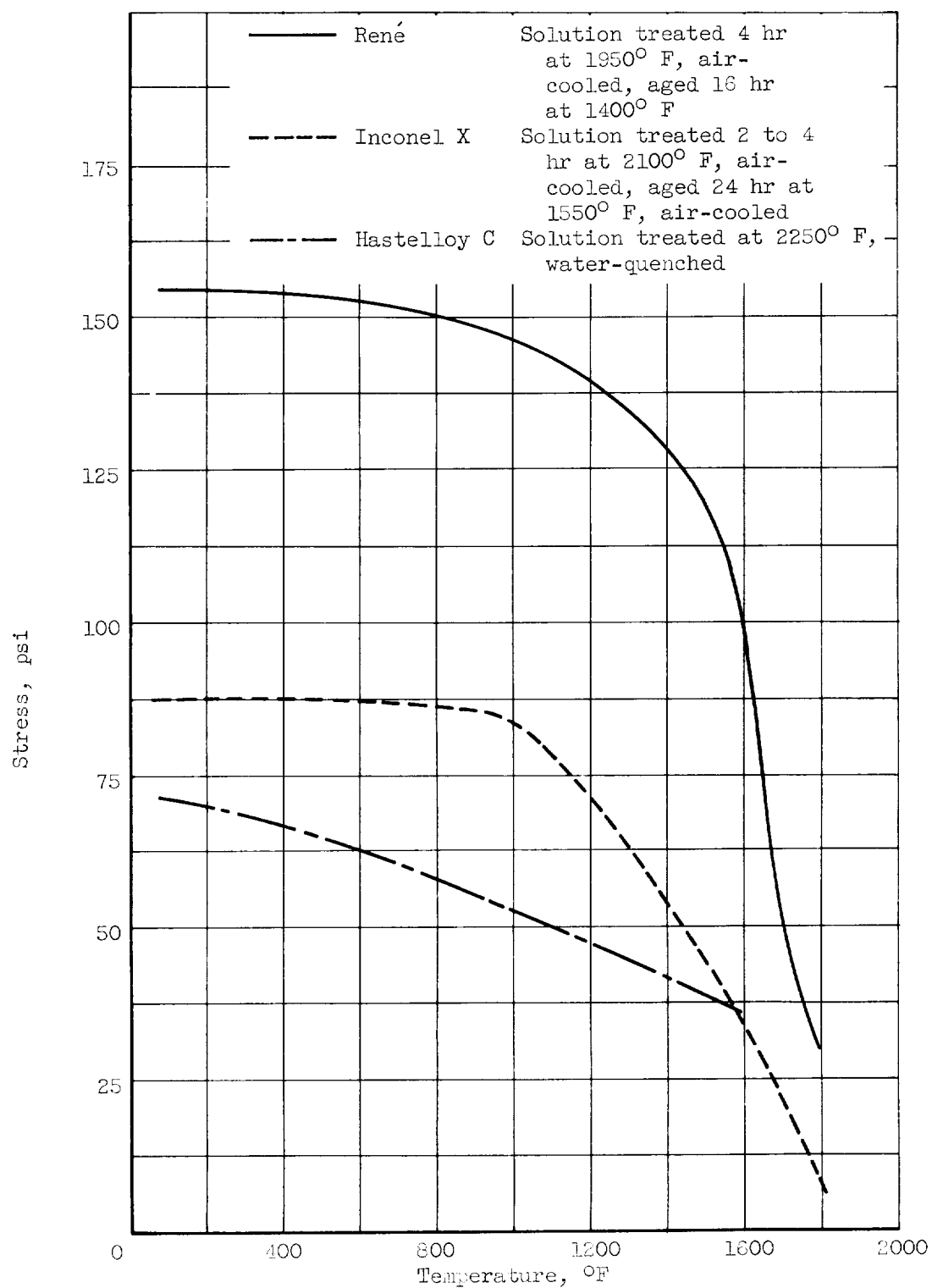
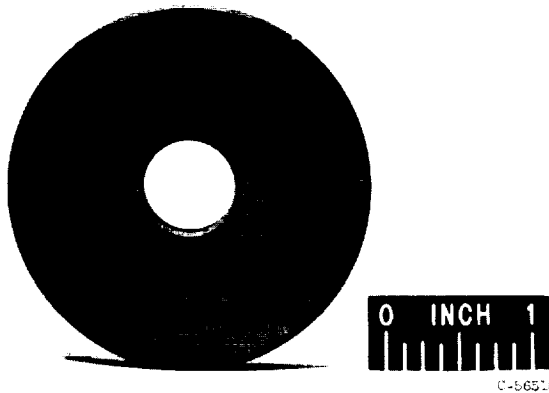
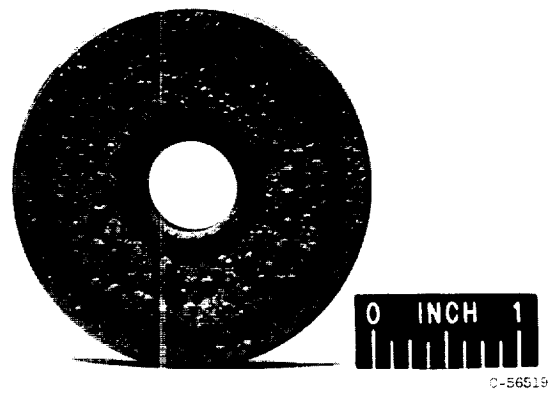


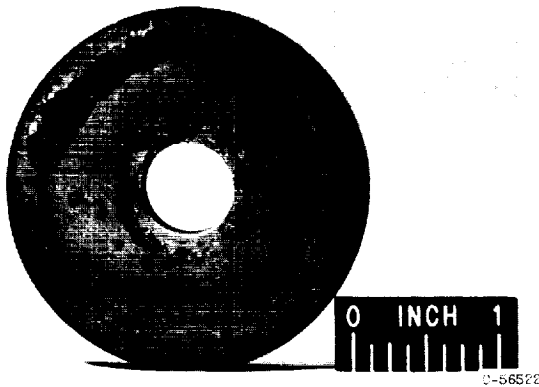
Figure 8. - Yield strength (0.2-percent offset) of alloys used in this investigation. (Data from ref. 11.)



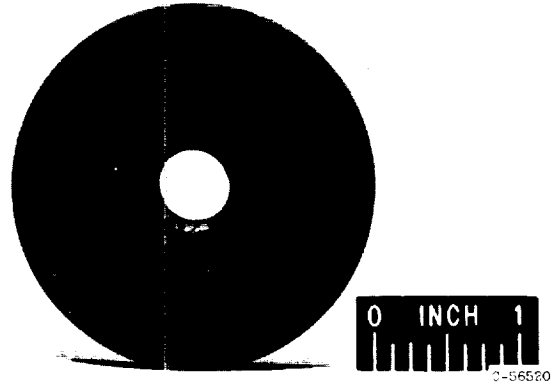
(a) Coating on Inconel X after 345 minutes at 1500° F.



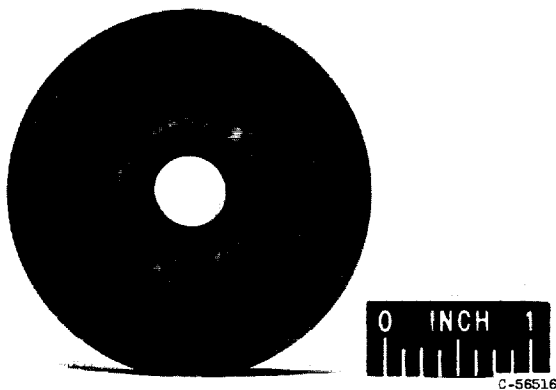
(b) Coating on Inconel X after 230 minutes at 1600° F.



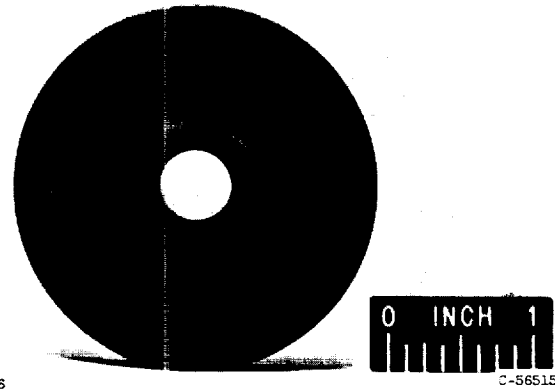
(c) Coating on Hastelloy C after 42 minutes at 1600° F.



(d) Coating on René 41 after 390 minutes at 1700° F.



(e) Coating on René 41 after 315 minutes at 1800° F.



(f) Coating on René 41 after 70 minutes at 1900° F.

Figure 10. - Influence of alloy selection on adhesion of ceramic-bonded calcium fluoride after exposure to elevated temperatures.

E-1401

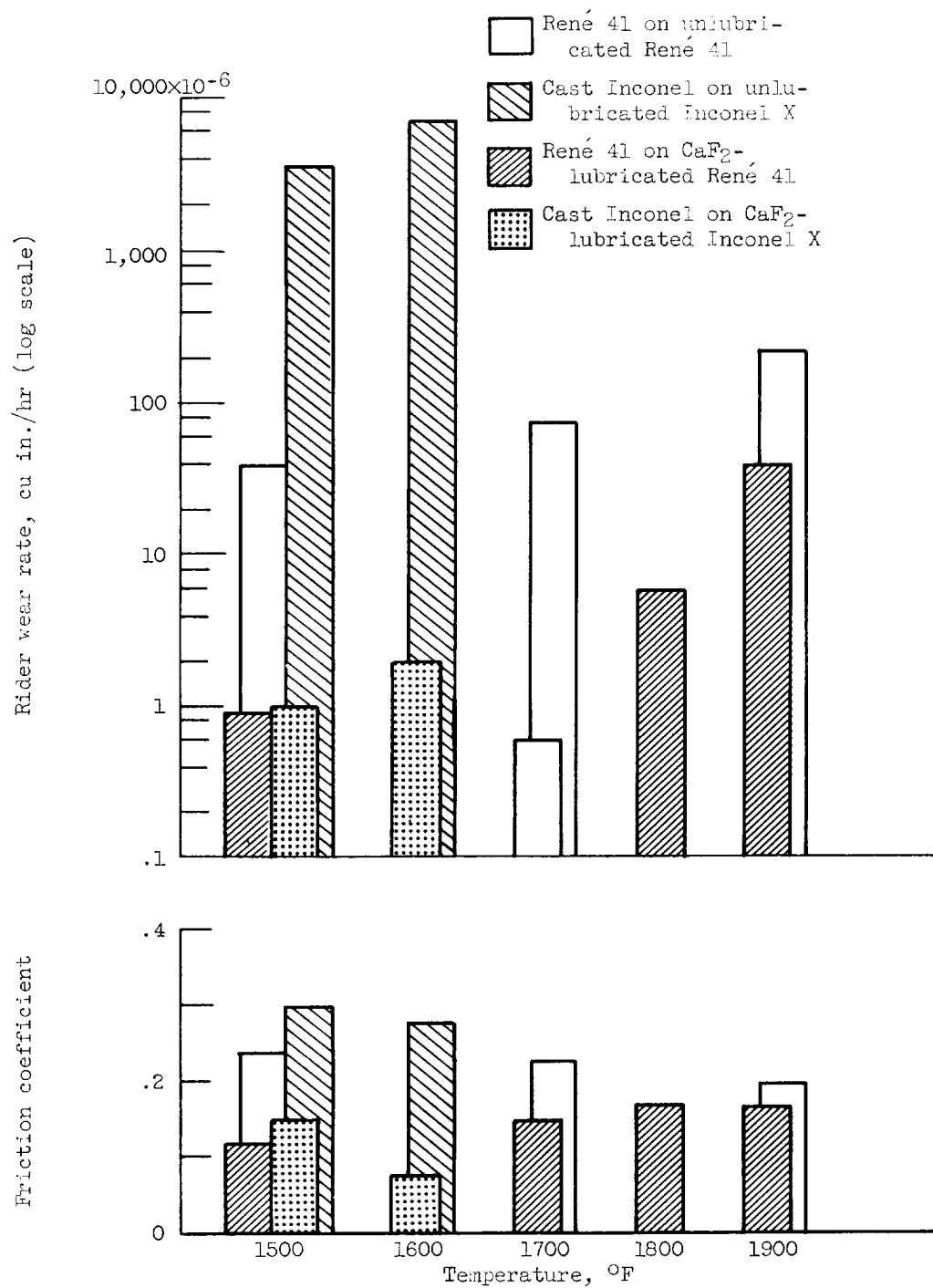
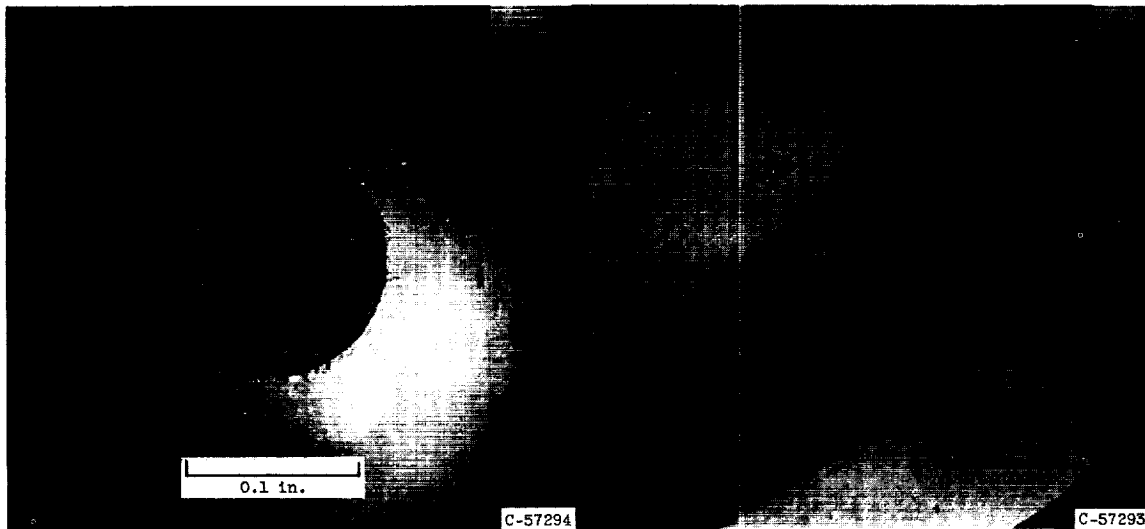
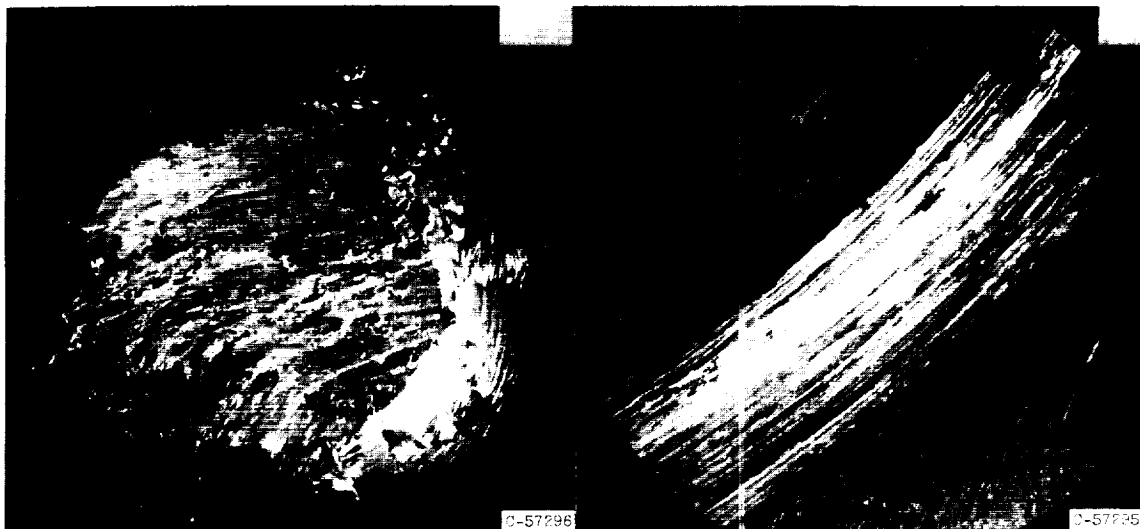


Figure 11. - High-temperature friction and wear properties of unlubricated René 41 and Inconel compared with calcium fluoride - lubricated metals. Atmosphere, air; rider specimens, 3/16-inch-radius hemispheres; sliding velocity, 600 feet per minute; load, 1000 grams.



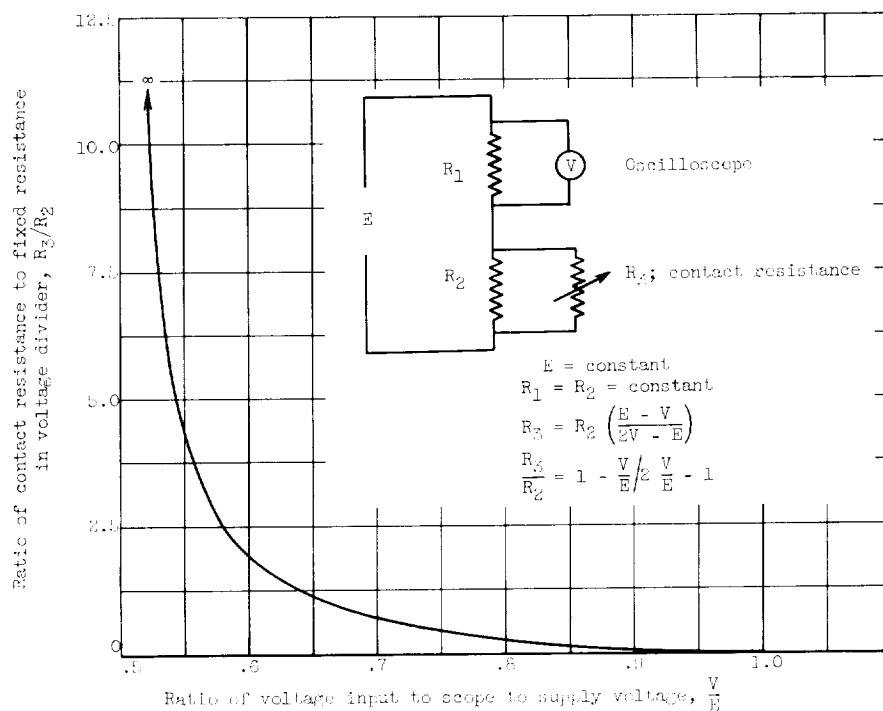
(a) Run in air for 1 hour; friction coefficient, 0.23.



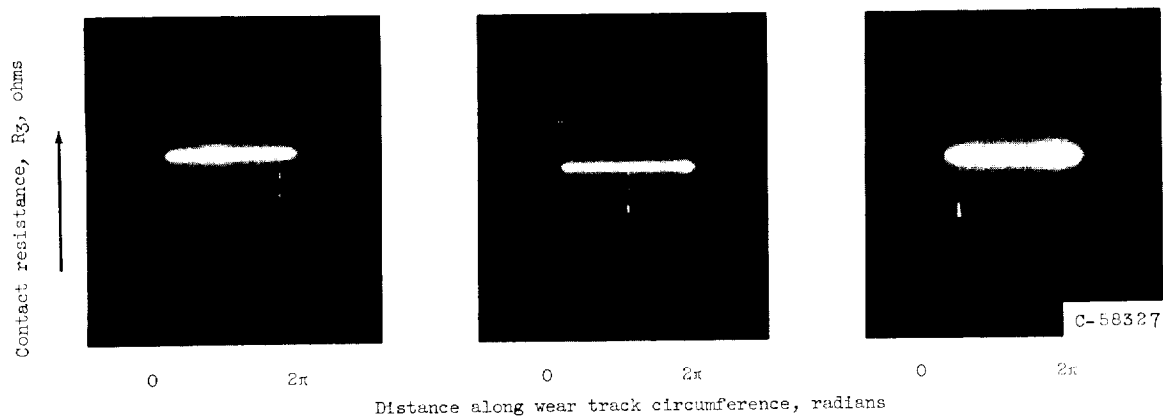
(b) Run in 90-percent-nitrogen - 10-percent-hydrogen atmosphere for 50 minutes; friction coefficient, 0.40.

Figure 12. - Surface damage and friction coefficients of René 41 in air and in reducing atmosphere at 1700° F. Rider specimens, 3/16-inch-radius hemispheres; sliding velocity, 600 feet per minute; load, 1000 grams.

E-1401



(a) Voltage across oscilloscope as function of contact resistance between slider specimens.



(b) Oscilloscope test patterns demonstrating that spikes in scope traces correspond to definite angular positions on wear track. At left, insulating coating was intentionally shorted out about 350° from arbitrary zero point; in center, at 180° ; and at right, at about 10° .

Figure 13. - Circuit characteristics used to measure electrical resistance distribution along circumference of wear track generated on surface of insulating coating.

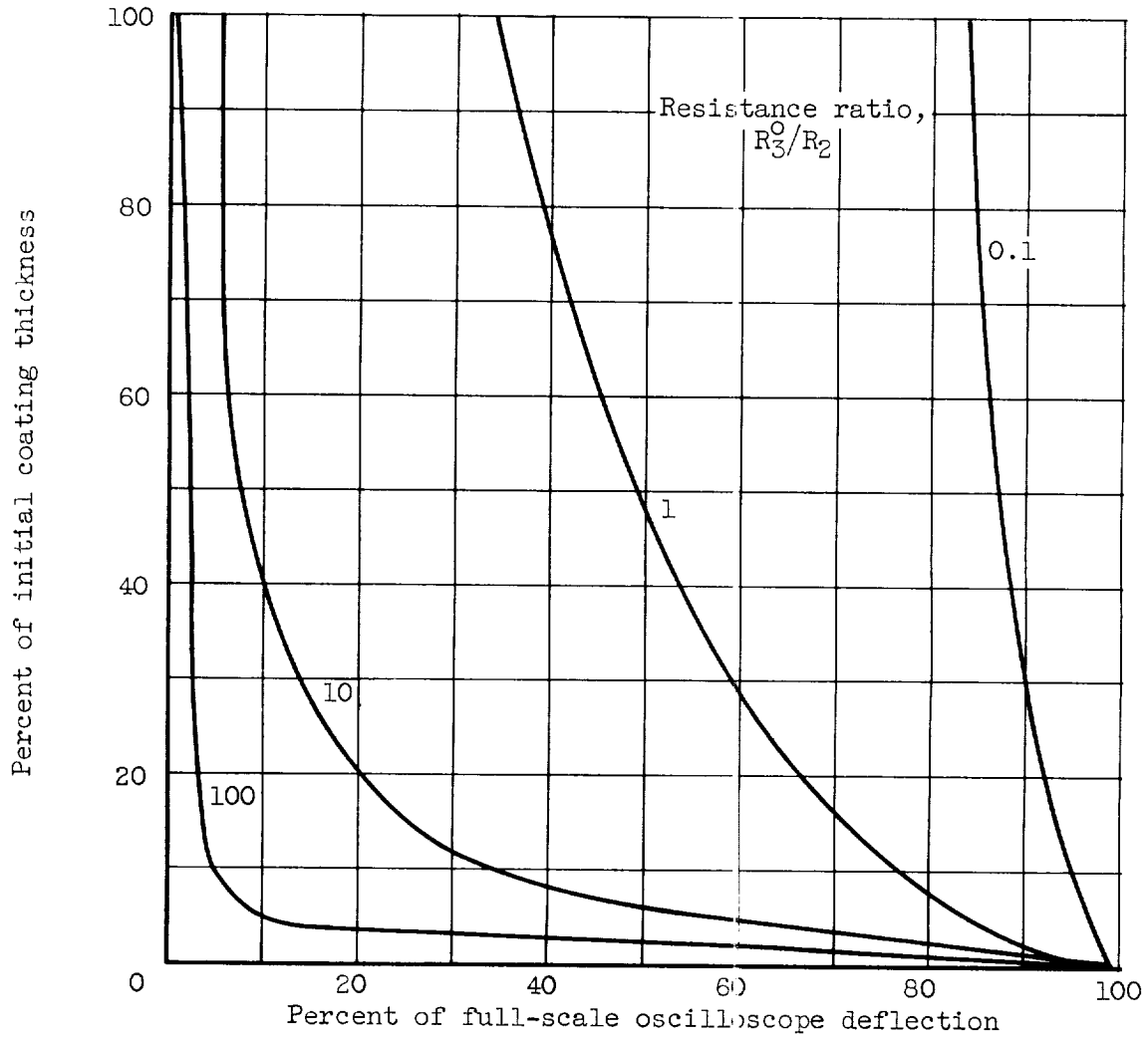


Figure 14. - Coating thickness as function of oscilloscope deflection for circuit of figure 13. Family of curves demonstrates how sensitivity of measurements is influenced by ratio of initial coating resistance to fixed resistance R_3^0/R_2 .

E-1401

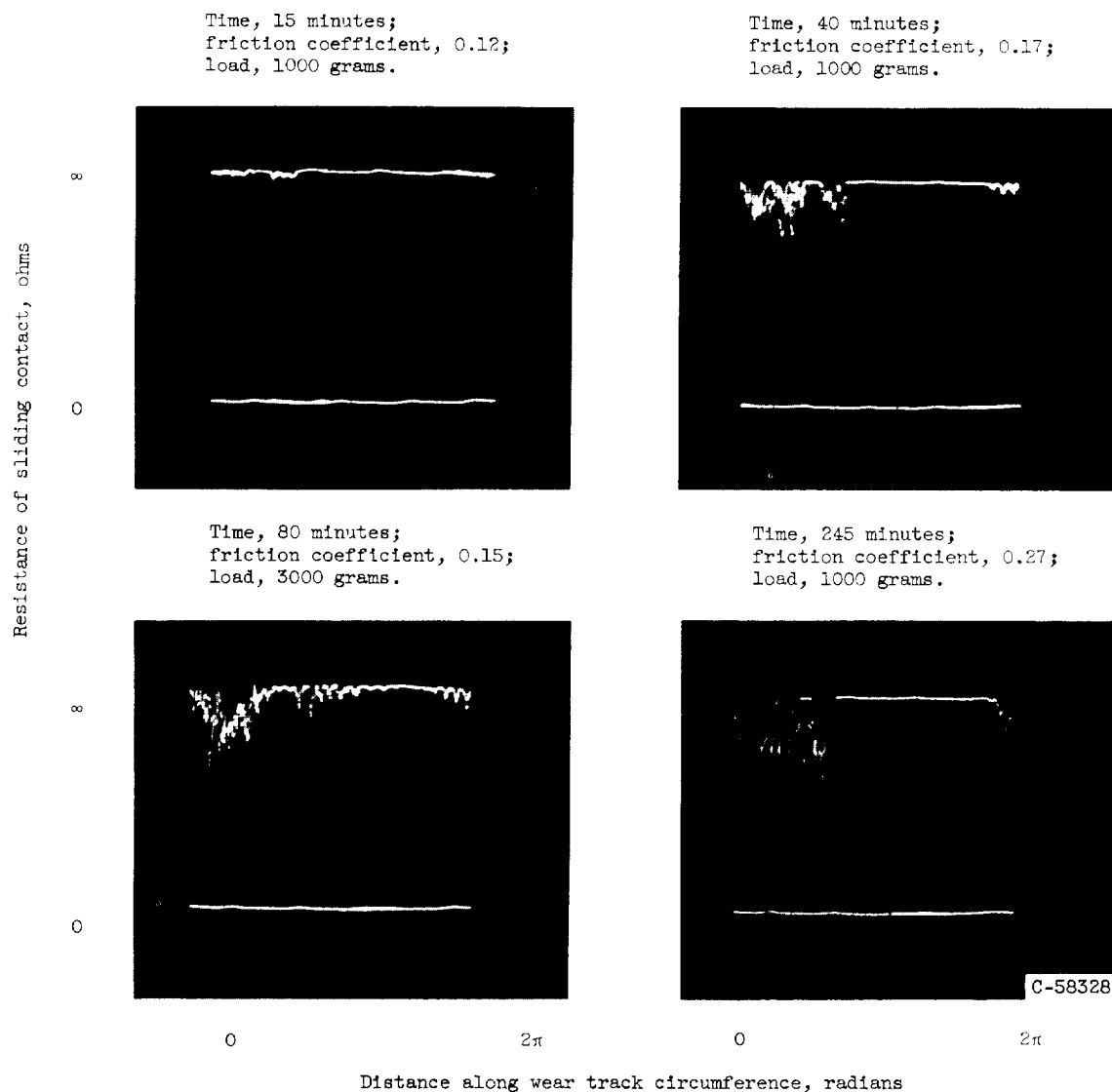
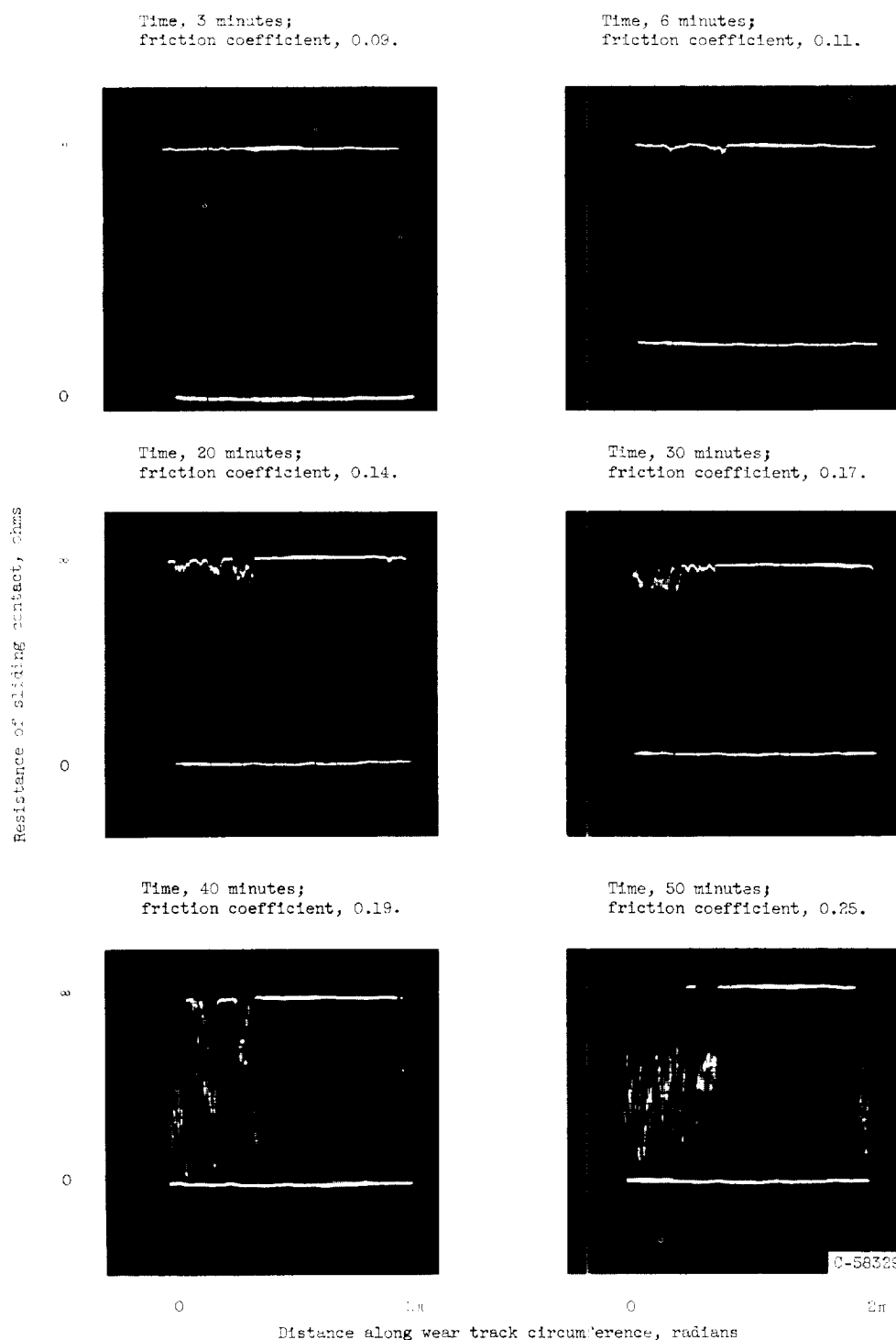


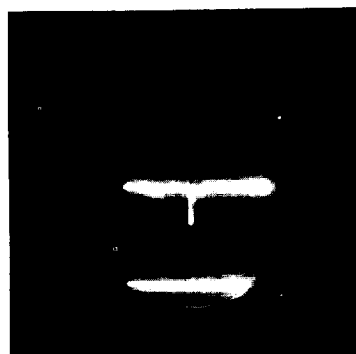
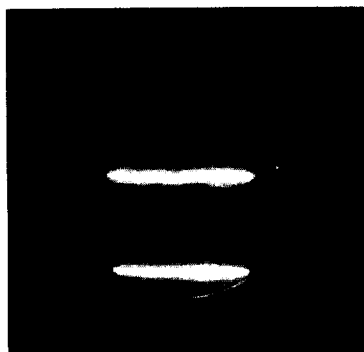
Figure 15. - Oscilloscope traces of electrical resistance distribution along circumference of wear track. Series corresponds to progressive failure of enamel-bonded calcium fluoride coating on preoxidized Inconel X with overlay. Temperature, 1000° F; coating thickness, 0.001 inch; rider specimens, 3/16-inch-radius hemispheres; sliding velocity, 430 feet per minute; resistance ratio, $R_3^0/R_2 = 10$.



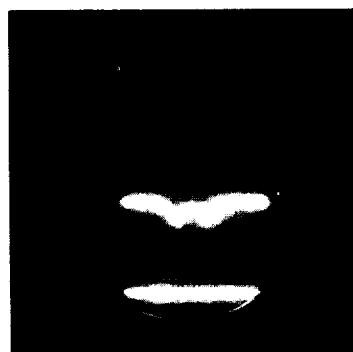
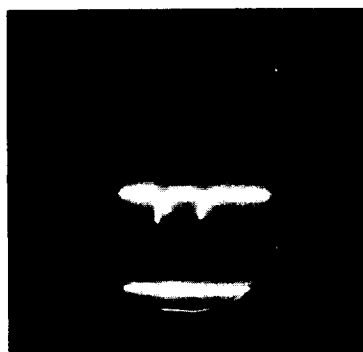
(a) Temperature, 500° F.

Figure 16. - Oscilloscope traces of electrical resistance distribution along circumference of wear track. Series corresponds to progressive failure of lead silicate-bonded lead monoxide solid lubricant coating on 440-C stainless steel. Coating thickness, 0.001 inch; rider specimens, cast Inconel (3/16-inch-radius hemispheres); sliding velocity, 430 feet per minute; load, 1000 grams; resistance ratio, $R_3/R_2 = 10$.

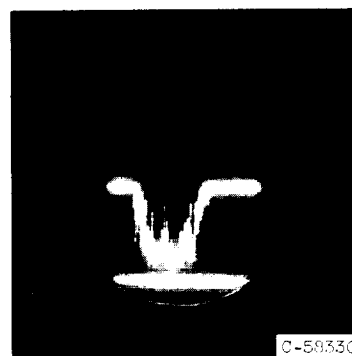
Time, 15 minutes;
friction coefficient, 0.11.



Time, 27 minutes;
friction coefficient, 0.09 to 0.12.



Time, 118 minutes;
friction coefficient, 0.24 to 0.28.

 2π

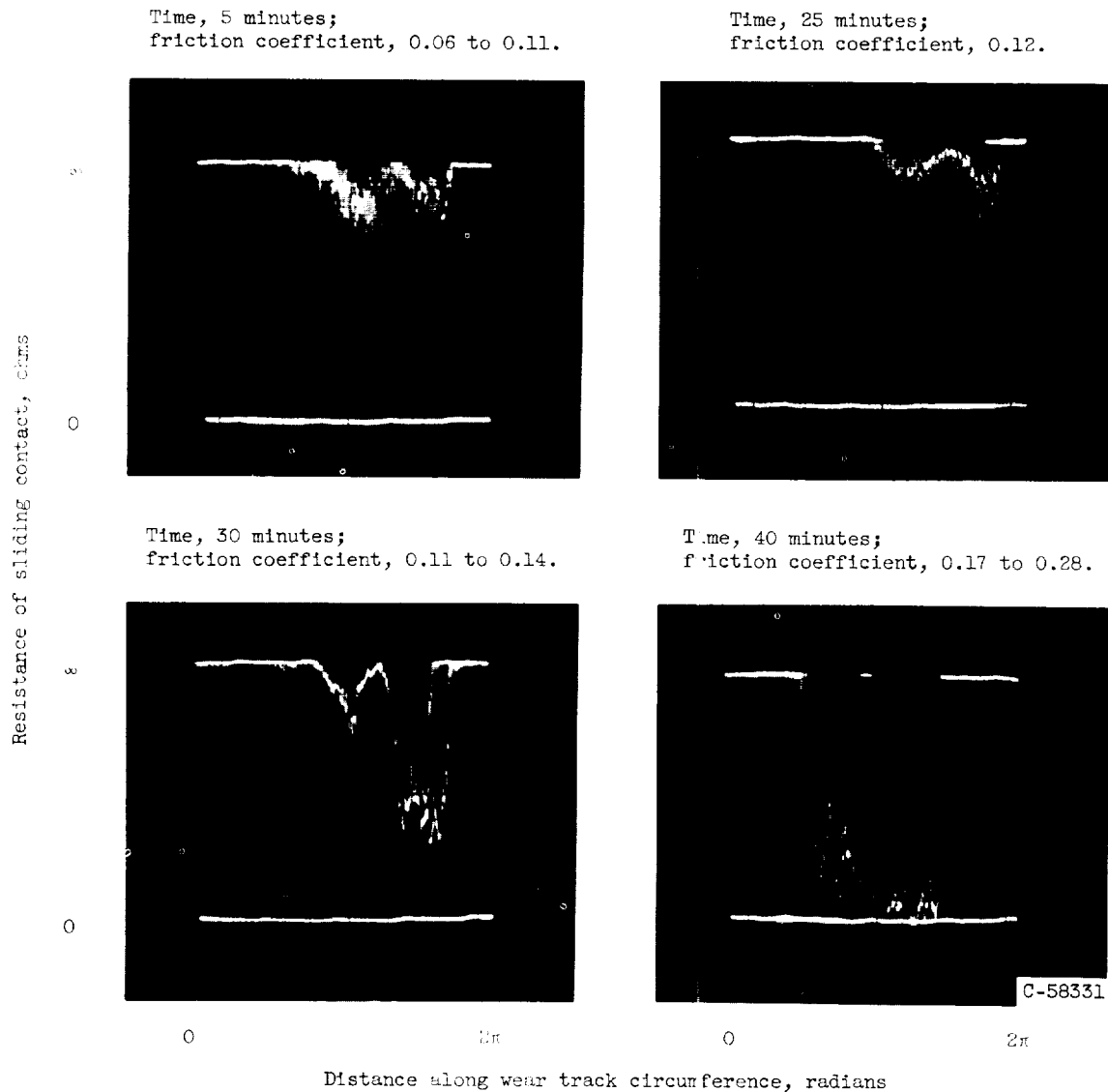
C

 2π

C-59330

(b) Temperature, 1000° F.

Figure 16. - Continued. Oscilloscope traces of electrical resistance distribution along circumference of wear track. Series corresponds to progressive failure of lead silicate-bonded lead monoxide solid lubricant coating on 440-C stainless steel. Coating thickness, 0.001 inch; rider specimens, cast Inconel (3/16-inch-radius hemispheres); sliding velocity, 430 feet per minute; load, 1000 grams; resistance ratio, $R_2/R_0 = 10$.



(c) Temperature, 1250° F.

Figure 16. - Concluded. Oscilloscope traces of electrical resistance distribution along circumference of wear track. Series corresponds to progressive failure of lead silicate-bonded lead monoxide solid lubricant coating on 440-C stainless steel. Coating thickness, 0.001 inch; rider specimens, cast Inconel (3/16-inch-radius hemispheres); sliding velocity, 430 feet per minute; load, 1000 grams; resistance ratio, $R_3/R_2 = 10$.

E-1401

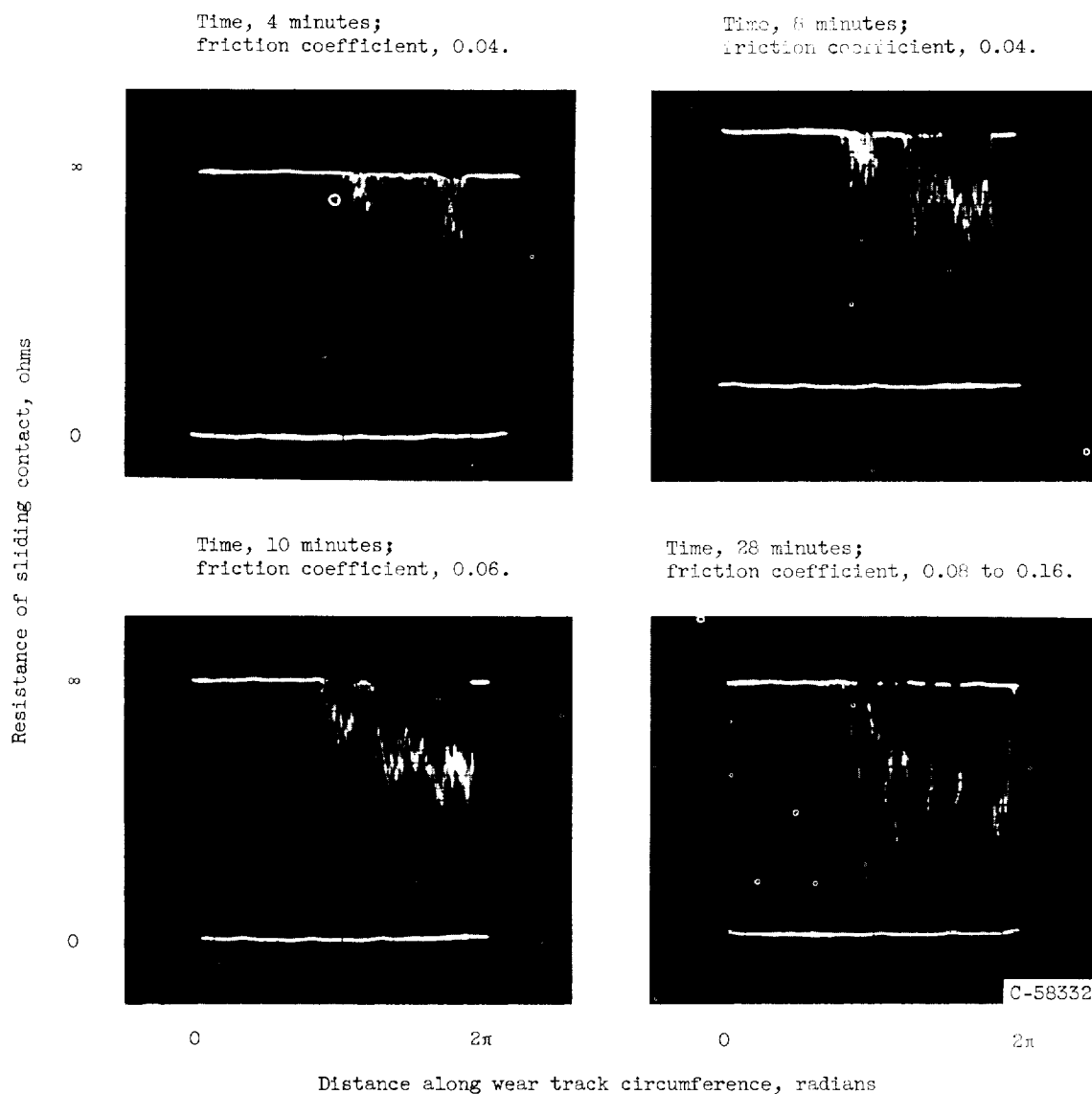


Figure 17. - Oscilloscope traces of electrical resistance distribution along circumference of wear track. Series corresponds to progressive failure of resin-bonded molybdenum disulfide coating on Inconel X. Temperature, 500° F; coating thickness, 0.0002 inch; rider specimens, cast Inconel (3/16-inch-radius hemispheres); sliding velocity, 430 feet per minute; load, 1000 grams; resistance ratio, $R_2^0/R_1 = 1$.

